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The topical conference was held in the Colorado Convention Center and was highly successful. There were some 59 papers (including 18 invited papers) presented in the Topical Conference. Of these, 52 papers were eventually published as full length articles. Abstracts of the papers presented are given in this report. The papers that were published are in Journal of Vacuum Science and Technology B, Volume 13, Number 4, pages 1862-1928, July/Aug 1995.

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AMERICAN VACUUM SOCIETY

120 WALL STREET, 32ND FL., NEW YORK, NY 10005 • Fax 212/248-0245

FINAL REPORT

ARPA/AFOSR CONTRACT #F49620-94-1-0441

Topical Conference on Manufacturing Science and Technology

This is the final report for ARPA/AFOSR Contract #F49620-94-1-0441, to put on a topical conference on Manufacturing Science and Technology the week of October 24-28, 1994 in Denver, Colorado, in conjunction with the 41st National Symposium of the American Vacuum Society.

The topical conference was held in the Colorado Convention Center and was highly successful. There were some 59 papers (including 18 invited papers) presented in the Topical Conference. Of these, 52 papers were eventually published as full length articles. Abstracts of the papers presented are given in this report. The papers that were published are in Journal of Vacuum Science and Technology B, Volume 13, Number 4, pages 1862-1928, July/Aug 1995.

The Topical Conference was well attended, and we are very pleased with both the number of papers presented and their quality. There is no attendance list because the Topical Conference was part of the overall Symposium, and there was no separate registration.

The American vacuum Society is extremely interested in encouraging and improving our Conferences on Manufacturing Science and Technology. We feel that we are a very interdisciplinary society, having major efforts in basic research, applied research and development, manufacturing, as well as in marketing and management. We also cover areas of physics, chemistry, engineering, and biology. Our aim is to facilitate and promote the interaction between science and technology and manufacturing, and to provide a mechanism for transferring methods and results of science and technology into manufacturing, and to enable scientists and engineers to be cognizant of requirements of manufacturing. These goals are very important to our national economy and industrial support as well as to the society. We are cooperating with members of IEEE in organizing and supporting these Topical Conferences, as well as with members of Sematech.

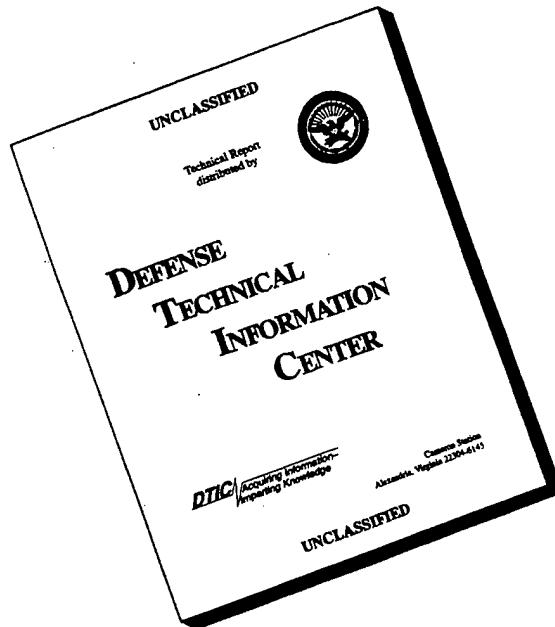
AVS also held a Topical Conference on this subject in Minneapolis, Minn. in October of 1995, and plans are under way to have the Conference in October, 1996 in Philadelphia, PA - again in conjunction with the Annual Symposium of the American Vacuum Society.

Enclosed are a listing of abstracts of the papers presented. Also enclosed is a listing of the papers that were published in JVST B that originated at this conference. The American Vacuum Society certainly thanks ARPA and the Air Force Office of Scientific Research for making it possible to put on this Topical Conference.



N. Rey Whetten
Principal Investigator
AVS Technical Director

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MANUFACTURING SCIENCE AND TECHNOLOGY

COLORADO CONVENTION CENTER

DENVER, COLORADO

24-28 OCTOBER 1994

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AMERICAN VACUUM SOCIETY

41st National Symposium

and

*NANO 3: THIRD INTERNATIONAL CONFERENCE
ON NANOMETER-SCALE SCIENCE & TECHNOLOGY*

*Colorado Convention Center
Denver, Colorado
October 24-28 1994*

FINAL PROGRAM

VM-ThM	Pulsed Laser and Pulsed Ion Technology for Film Deposition and Surface Modification <i>L. Greer, Raytheon; D. McIntyre, Sandia Nat. Labs</i>	Code	Session
VM-ThA	Manufacturing Technology for Coatings <i>K. Legg, Northwestern Univ.; D. Glocker, Eastman Kodak; P. LeFebvre, OCLI</i>	VT-MoA	Gas Flow, Partial Pressure Analysis, and Leak Detection <i>M. Boeckmann, Vacuum Technology, Inc.; A. Fine, EPA</i>
VM-FrM	Surface Engineering for Wear and Corrosion Protection <i>A. Matthews, Univ. Hull</i>	VT-TuM	Vacuum Systems for Accelerators and Fusion <i>P. LaMarche, Princeton Plasma Phys. Lab; R. Wavrick, Sandia Nat. Lab.</i>
		VT-TuA	Vacuum System Outgassing and Cleaning <i>S. Tison, NIST</i>

Vacuum Technology Division

The 1994 Vacuum Technology Division program covers vacuum science and technology issues that are important to all users of vacuum equipment and processes. These topics include fundamentals of the production, measurement and control of the vacuum environment.

The program begins Monday morning with a session on total pressure measurement. Characterization of any system or process usually requires a reliable measurement of the pressure. This session presents pressure gauge calibration and reliability measurements and several promising new measurement methods. An invited paper and several contributed papers highlight silicon-based micro sensors. Partial pressure, gas flow, and leak detection methods are covered in Monday afternoon's session. Invited papers cover gas sampling techniques and the development of advanced vacuum insulation for use in energy conservation.

New to the Vacuum Technology Division program will be a technical poster session on Monday afternoon covering many aspects of vacuum science and technology. This session will provide excellent opportunity for questions and discussions with the presenters.

The large national and international facilities for particle physics and fusion research require complicated and high performance vacuum systems. Tuesday morning's session describes progress in the specialized materials, vacuum components, and vacuum vessel conditioning techniques developed for these large facilities that often find use in smaller systems for research and manufacturing. Invited talks will summarize the D-T experiments at Princeton which recently produced world's record fusion power with the TFTR tokamak, and the planned design of a new National Ignition Facility for inertial fusion research.

The understanding of the source of gas phase and surface contaminants is important for the design and operation of vacuum systems where impurity control is a critical parameter. The VTD's Focus Session on contamination control leads off with an invited talk by S. Tison from NIST who will summarize a recently held workshop sponsored by the Division on the measurement and control of water in vacuum systems. Related papers will discuss novel surface preparation methods and conditioning techniques which minimize thermal outgassing. The session concludes with a cluster of papers discussing new developments in vacuum materials for use in the next generation of storage rings for minimizing photodesorption gas loads.

Topical Conferences

Manufacturing Science and Technology

Building on last year's successful Topical Conference on manufacturing, and as a prelude to the recent establishment of a new Manufacturing Science and Technology Group within the AVS, this Second Topical Conference will comprise 3 1/2 days of technical sessions. The technical program of the Topical Conference has been jointly planned with the IEEE, which is a Technical Co-Sponsor.

The Topical Conference, focusing primarily on the microelectronics industry, is aimed at elucidating the scientific and technical issues which underpin effective manufacturing. An **Overview** session of primarily invited speakers will highlight the role of equipment, future manufacturing concepts, research opportunities, and environmental issues.

Two sessions are devoted to **Advanced Manufacturing Equipment** for both thermal and plasma processes, underscoring the importance of the equipment industry to microelectronics manufacturing. The equipment industry represents a critical component to microelectronics manufacturing competitiveness, and the AVS hopes to contribute significantly in this area, building on its scientific strengths and on the long-standing alliance between its university, government, and industrial members.

A session on **Diagnostics, Sensors, and Control** represents a crucial element for manufacturing and also draws on a traditional strength of the AVS; this session is part of a multi-session focus for the entire National Symposium. The session on **Micro-Contamination and Defects**, part of different multi-session focus in the meeting on surface contamination and control, will address particulate and molecular contamination, defects, and their relation to equipment and process. The **Process and Equipment Modeling** session will include an invited talk on the role of equipment modeling and process synthesis, as well as talks on both reactor-scale and microfeature-scale issues in chemical processes.

A joint session on **Vacuum Process Control** for manufacturing will emphasize aspects of vacuum technology with major manufacturing impact in microelectronics, particularly process and equipment control, ergonomics, and diagnostic sensors. Finally, the Topical Conference is co-sponsoring other sessions in the **Symposium on Industrial Applications of Scanning Probe Microscopy** and on **Surface Preparation and Passivation**.

Code	Session	Code	Session
MS-MoA	Process and Equipment Modeling <i>Z. Lemnios, ARPA</i>	BI-MoA	Cell-Solid Surface Interactions <i>J. Davies, Univ. Toronot; D. Okrongly, Appl. Immune Sci.</i>
MS-TuM	Manufacturing Overview and Environmental Issues <i>S. Harrell, Sematech; G. Pitts, MCT Corp., R. Kerby, EPA; D. Herr, Semicond. Res. Corp.</i>	BI-TuM	Protein-Solid Surface Interactions <i>P. Stayton, Univ. Washington; R. Marchant, Univ. Washington</i>
MS-TuA	Advanced Manufacturing Equipment - A <i>R. Bachrach, Appl. Materials; E. van de Ven, Novellus; K. Okomura Toshiba</i>	BI-TuA	The Biosensor-Biology Interface <i>M. Meyerhoff, Univ. Michigan; F. Ligler, Naval Res. Lab; D. Charych, Lawrence Berkeley Lab.</i>
MS-WeM	Advanced Manufacturing Equipment - B <i>R. Wright, Sematech; G. Selwyn, IBM</i>	BINS-WeM	Artificial Cellular Assemblies <i>T. Matsuda, Nat. Cardiovas. Res. Ctr. Japan</i>
MS-WeA	Diagnostics, Sensors, and Control <i>S. Butler, Texas Instr.; J. O'Neill, IBM; J. Wiczer, Sandia Nat. Lab.</i>		
MS-ThM	Micro-Contamination and Defects <i>B. Hermsmeier, IBM; H. Walker, Texas A&M Univ.</i>		
MSVT-ThA	Vacuum Process Control for Manufacturing <i>D. Miller, Sandia Nat. Lab.; R. McMahon, Techware</i>		

Biomaterials Interfaces

The Second Biomaterials Interfaces Topical Conference has been organized to provide a special forum where biologists and interface scientists can share their unique perspectives to the R&D opportunities in biomaterials. This mixing of disciplines is important because many biological phenomena occur at the solid-liquid interface. Two sessions are devoted to the interactions of proteins and cells with solid-state interfaces. The perspectives of the speakers in these two sessions range from fundamental interactions to developed applications. A third session addresses the issues involved in sensors' interactions with biological systems, from design to applications. The fourth session, which is new to the Conference this year, described progress in the field of artificial cellular assemblies. The dominant theme in this session is the creation of artificial neuronal networks and their characterization. Most of the top groups in the world involved in this exciting frontier research are represented.

The conference also includes three joint sessions with the NANO 3 Conference. Two sessions deal with Biology at the Nanoscale, a collection of work that features applications of recent breakthroughs in Scanning Tunneling Microscopy/Spectroscopy, Force Microscopy, Near Field Optical Microscopy, and related technology. The investigation of DNA binding interactions is highlighted in one of these sessions. The final joint session lists developments in proximal probes, including medical and neuronal devices. The NANO 3 Conference also features related work in surface modification, nanomechanics, and nanotribology. In all, the combination of sessions give a good overview of the exciting area of biological-solid state interactions, with direct impact on human health issues and biotechnology.

Focus Areas

"Focus Areas" are a new concept we are introducing at this meeting in order to highlight new technical areas that will likely be important topics at future symposia, and to highlight areas of common interest among a number of different divisions. We have scheduled sessions within each focus area to minimize conflicts, so that the attendee can obtain diverse perspectives from a number of AVS Divisions. The Focus Areas and sessions included in them are listed below. These sessions are also highlighted on the "Program at a Glance" in the rear of the program book.

Si-based Optoelectronics

Sponsored primarily by the Electronic Materials and Processing Division, talks include materials, structures and devices for optoelectronics, based on silicon technology, including waveguides, light emitters, interconnects, and system applications.

Code	Session
EM-TuA	Silicon-based Optoelectronics
EM-WeM	Heterostructures for Optoelectronics
EM-WeA	Wide-bandgap Nitrides
NS2-WeA	Optical Properties of Silicon Nanostructures

Sensors, *in-situ* Diagnostics and Process Control

This broadly defined area includes sessions on sensor materials and devices, use of sensors and other diagnostics in the process environment, and control of processes using *in-situ* diagnostic information.

Code	Session
SS1-MoA	Surface Mechanisms and Materials for Chemical Sensors
TF-TuM	Thin Films for Sensors
BI-TuA	The Biosensor-Biology Interface
PS-WeM	Plasma Diagnostics
MS-WeA	Diagnostics, Sensors, and Control

discussed. The technique has recently been used to show that transverse momentum is largely conserved for electron transport across the commensurate $\text{NiSi}_2/\text{Si}(111)$ interface, whereas it is not conserved for incommensurate metal-semiconductor systems. The energy dependence of elastic and inelastic mean free paths of hot electrons in Pd layers deposited on $\text{Si}(100)$ and (111) substrates has been obtained as well. BEEM can be used to measure directly the quantum yield of electron-hole pair generation by impact ionization of injected hot electrons, which will be discussed for Si. Of current interest is the characterization of transport properties of thin insulating layers (2-6 nm) placed between the metal and the semiconductor. Transport takes place mainly through electron injection into the conduction band of the insulator. The application of a bias across the insulator provides an additional variable to study the de-excitation processes of hot carriers in the conduction band of the insulator. Recent results for SiO_2 interlayers will be presented.

INVITED

4:00 pm EM-MoA7 Epitaxy in the Room Temperature Reaction of Ni on Si(111) Studied with Synchrotron X-ray Diffraction, P. Yang, M. Y. Lee and P. A. Bennett, Physics Dept., ASU, Tempe, AZ 85287-1504, P. J. Eng, AT&T Bell Labs, Murray Hill, NJ 07974 and I. K. Robinson, Physics Dept., UI, Urbana, IL 61810.

We find that deposition of Ni on $\text{Si}(111)-7 \times 7$ at room temperature produces a commensurate, epitaxial silicide at the buried metal/silicon interface. Intensity oscillations occur signifying a layerwise etching of the substrate, similar to the Pd/Si case.[1] Model calculations of the intensity profile along several crystal truncation rods allows a clear structure assignment as Ni_2Si -theta. For the first several Å, the integrated intensities in the Ni_2Si reflections account for all the deposited metal, ie: no other phase forms. The structure factors show a Debye-Waller like variation against Q with an extremely large atomic displacement $u_{\text{rms}} \sim 0.4$ Å. Diffuse intensity appears throughout the Brillouin zone, suggesting a high density of point defects. The nature of disorder in the silicide film explains why epitaxy has not been seen at room temperature using other techniques such as MEIS, LEED, etc. Implications for low temperature epitaxial growth are discussed.

[1] P. A. Bennett, B. Devries, I. K. Robinson and P. J. Eng, Phys. Rev. Lett. 69, 2539 (1992).

4:20 pm EM-MoA8 Phonon Scattering of BEEM Electrons at Au/Si(100) Schottky Interfaces, C. A. Ventrice, Jr., V. LaBella, G. RamaSwamy, and L. J. Schowalter, Rensselaer Polytechnic Institute, Troy, NY 12180 U.S.A.

The relative importance of temperature dependent scattering mechanisms (i.e., acoustic phonon scattering within the metal overlayer and optical phonon scattering within the semiconductor) on ballistic electron emission microscopy (BEEM) electron transport across Au/Si(100) Schottky interfaces is not well understood. To better quantify the effects of phonon scattering, BEEM measurements on Au/Si(100) interfaces have been performed at both room temperature (RT) and 77 K for several Au overlayer thicknesses on n-type Si ($\rho = 2-4 \text{ ohm-cm}$). The onset of the BEEM collector current, which determines the Schottky barrier height, was measured to be 0.82 eV at 77 K and 0.78 eV at RT. The observed shift in the onset of the collector current results from both a narrowing of the band gap of Si at elevated temperatures and thermal broadening of the tip's Fermi function at RT. Measurements of the average BEEM electron transmittance for energies up to ~0.4 eV above the 0.8 eV Schottky threshold have revealed a reduction in BEEM electron transmittance at RT for each Au film thickness. The measured relative transmittance at RT to that at 77 K ranged from 0.55 to 0.68 for Au overlayer thicknesses of 67 Å to 140 Å. Calculations of the temperature dependent attenuation length within the Au overlayer have indicated that multiple reflections of the BEEM electrons within the Au overlayer must be taken into account to explain the observed functional dependence of the BEEM electron transmittance. (This research was supported in part by the U.S. Air Force and the Office of Naval Research).

4:40 pm EM-MoA9 Metal (CoSi_2)/Insulator (CaF_2) Resonant Tunneling Diodes and Transistors, M. Asada, M. Watanabe, T. Suemasu, and Y. Kohno, Tokyo Institute of Technology, Meguro-Ku, Tokyo 152, Japan.

Metal/insulator heterostructures can be considered attractive material for high-speed quantum electron devices, because high carrier density of metal and low dielectric constant of insulator are suitable for the size reduction and high-speed operation. Also, in certain material combination, strong quantum interference is expected due to

very large band discontinuity at the heterointerface (~10 eV), which can be utilized for multifunctional and high-transconductance devices.

In this paper, we report epitaxial growth of metal/insulator heterostructure and its application to quantum electron devices. CoSi_2 and CaF_2 were chosen for metal and insulator because of their small lattice mismatch to Si (~1.2 and +0.6%, respectively). CaF_2 was grown at low temperature (~450°C) by the partially ionized beam epitaxy to suppress the thermal damage of CoSi_2 underlayers. CoSi_2 was grown by the two-step growth of Si and Co to avoid island formation. By these technique, a few nanometer-thick $\text{CoSi}_2/\text{CaF}_2$ multilayer was obtained on $\text{Si}(111)$. Resonant tunneling diodes with two metal quantum wells showed negative differential resistance (NDR) at 77 K and room temperature. The change of the applied voltage at NDR with quantum well thickness agreed with theoretical calculation. Resonant tunneling transistors were fabricated by connecting the base electrode to one of the quantum wells of the resonant tunneling diode with high-selective wet chemical etching process. Transistor action with NDR was observed at 77 K. Depending on the choice of the two wells as the base, two different characteristics were obtained. Transfer efficiency close to unity was obtained for electrons from emitter to collector through the resonant levels in these transistors.

INVITED

MANUFACTURING SCIENCE AND TECHNOLOGY

Room A110 - Session MS-MoA

Process and Equipment Modeling

Moderator: K. Uram, Lam Research Corporation.

2:00 pm MS-MoA1 Flux Distributions and Growth Rate Uniformities from Hexagonal Collimators, Z. Lin and T. S. Cale, Center for Solid State Electronics Research, Arizona State University, Tempe, AZ 85287-6006.

We use the Monte Carlo method to simulate collisionless transport through collimator cells and collisional transport from the collimator to the substrate [1]. Three dimensional flux distributions are obtained for species exiting a single hexagonal collimator cell as functions of collimator aspect ratio, the sticking factor of the sputtered material in the collimator, and the flux distribution of moieties entering the collimator. We consider specular as well as diffuse re-emission, for species which have subunit sticking factors in the collimator. The larger the sticking factor and the higher aspect ratio, the higher the 'beaming' effect of the collimator and the smaller the fraction of moieties which make it through the collimator. Diffuse re-emission produces a more collimated beam than specular re-emission, but the fraction of the moieties which make it through the collimator is lower for diffuse re-emission.

After establishing the film growth rate profile on a flat wafer due to one collimator cell, local growth rates due to all of the cells are obtained by summing the fluxes from all contributing collimator cells. For a given collimator design, the variation in deposited film thickness is a function of collimator to wafer distance and sputter gas pressure. The longer the distance from the collimator to the wafer and the shorter the mean free path of the sputtered gas, the more collisions between molecules and the smoother the deposition profile. For hexagonal cells, the positions of the maxima in the deposition profile are under the corners of the cells, and the minima are under their middles.

1. D. Liu, S. K. Dew, M. J. Brett, *Thin Solid Films* 236, 267 (1993).

2:20 pm MS-MoA2 Modeling of Collimated Sputter-Deposition of Thin Films Using Molecular Dynamics: A Study of Step Coverage and Film Properties, C. C. Fang, State University of New York at Stony Brook, Department of Mechanical Engineering, NY 11794-2300, F. Jones, J. J. Hsieh, R. V. Joshi, IBM Watson Research Center, P.O. Box 218, Yorktown Heights, NY 10598, and V. Prasad, State University of New York at Stony Brook, Department of Mechanical Engineering, NY 11794-2300.

In the manufacture of sub-0.5 μm generation devices, the coverage of the liner on the bottom and the sidewall of the contact/via imposes severe limitations on the deposition process. Collimated sputtering is generally used to improve the coverage in high aspect ratio contacts and resolve several of the step coverage problems. Although this technique is widely used in electronics industry, the basic phenomena

associated with this process are poorly understood. This paper reports simulations of collimated sputter-deposition of thin films in micro-scale trench using a two-dimensional molecular dynamics model (MD) and examines the effects of process parameters on film properties and step coverage. The microstructure, intrinsic stresses and step coverage are calculated as a function of working pressure, power input, and aspect ratio of the collimator. The numerical results demonstrate that the non-unity stick coefficient strongly depends on the energetic neutral argon ejected from the target. The MD simulation technique is found to be helpful in optimization of the collimated sputter processes with high demands on uniformity and edge coverage. The simulation results for depositions in deep trenches are presented for a wide range of collimator aspect ratio, to explain the physics of the process.

2:40 pm MS-MoA3 Process and Equipment Modeling for Advanced Semiconductor Manufacturing, Zachary J. Lemnios, Program Manager, Advanced Research Projects Agency (ARPA).

Enormous development costs and excessive cycle times to transition new ideas into the manufacturing environment have limited semiconductor technology innovation. Through sequential and iterative process development, new process technologies require approximately 5 years to mature while process derivatives require 2-3 years to mature.

This paper addresses use of advanced process and equipment models to synthesize leading edge manufacturing tools. It also presents the use of these models in an integrated environment to synthesize of new manufacturing processes. This approach, will combine a new set of programmable factory tools with a set of embedded manufacturing models and computer aided design tools. The resulting architecture will provide the capability to concurrently design a circuit along with its associated process, significantly improving the yield learning curve for new products and technologies. The ability to make appropriate tradeoffs in performance, reliability, manufacturability, life cycle cost and cycle time will be possible with the availability of a process synthesis framework.

INVITED

3:20 pm MS-MoA5 Comparison of the Sandia DSMC Molecular Flow Model to Experiment¹, Paul K. Shufflebotham, Lam Research Corp., 4650 Cushing Pkwy., Fremont, CA 94538, Timothy J. Bartel, Sandia National Laboratories, Albuquerque, NM 87185-5800, Butch Berney, Lam Research Corp.

The Sandia DSMC rarefied gas dynamics model is used for the simulation of axisymmetric transition and molecular gas flow. This code has potential as a CAD tool for use in the vacuum design of low pressure plasma processing equipment, which typically operates in the transition flow regime. This assumes that the model is accurate and predictive. We have constructed an axisymmetric, transition flow, vacuum test cell (VTC) to test the model through direct comparison with experiment. The VTC was equipped with numerous capacitance manometers, a mass flow controller and a 2000 l/s compound magnetically levitated turbopump. Spatially-resolved pressure measurements were made at dry N₂ flows from 50 to 500 sccm. The inputs to the model were the VTC geometry and surface temperatures, input mass flow rates and output pumping speeds. Tens of thousands of computational particles were tracked for hundreds of thousands of time steps. After a brief discussion of the known model and experimental uncertainties, the capability of the DSMC code to predict the absolute values, flow rate dependencies and spatial variations of the pressures measured in the VTC will be described in detail. The utility of the model as a vacuum system CAD tool will also be briefly discussed.

¹71.

Work supported by Lam Research, Sematech and DOE.

3:40 pm MS-MoA6 Modeling of Ion Flux Uniformity in Radio Frequency Discharges: Effect of Electrode Topography, M. Dalvie, M. Surendra, G. S. Selwyn, C. R. Guarnieri, IBM T. J. Watson Research Center, POB 218, Yorktown Heights, NY 10598.

Plasma process non-uniformity is a major cause of yield loss during integrated circuit fabrication. Correlation of yield with chip placement on the wafer shows that yield falls off near the edge of the wafer, leading to an "edge exclusion" zone on the wafer. One possible cause of this is a non-uniformity in the ion flux. This is studied here by means of a 2-d self-consistent fluid model of a radio frequency discharge. Time-dependent electron and ion continuity, ion momentum balance, and electron energy balance equations are solved along with Poisson's equation. A semi-implicit time integration scheme and Galerkin Finite elements are used to solve the equations.

The simulation domain includes electrode topography that simulates

clampings and wafer edges. Results indicate that electrical property variation (e.g., conducting wafer to non-conducting clamping) causes the ion flux to fall off near the wafer edge. This may be partially responsible for the commonly observed edge exclusion on plasma processed wafers. Effect of the topography is related to plasma parameters, e.g., pressure, through the sheath thickness. Under high pressure (low sheath thickness) conditions, the ion flux is more sensitive to the topographical perturbation due to ionization rate enhancement in the corner. At low pressure, electrical property variation is more important. We also compare uniformity predictions obtained from the complete ion momentum balance to those from the drift-diffusion approximation (no ion inertia).

4:00 pm MS-MoA7 Simulation of a Tungsten Filled Via Process Module for Process Integration, D. S. Bang, K. Hsiao, J. P. McVittie, and K. C. Saraswat, Integrated Circuits Lab, Stanford University, CA 94303, Zoran Krivokapic, Advanced Micro Devices, P.O. Box 3453, Sunnyvale, CA 94088.

Tungsten is widely used for sub-micron VLSI via plugs because of the gap fill advantages of W-CVD over Al-PVD. A typical W-plug process contains multiple etching and deposition steps whose interactions become increasingly important in determining via characteristics as dimensions shrink. The use of computer simulation to examine equipment and process trade offs for an integrated Tungsten filled via process is demonstrated. Previous work in deposition and etching modeling has centered on developing topography models for specific process steps and equipment. This paper presents the development of modeling multistep fabrication processes for VLSI metallization. The interactions between different process steps during the fabrication of sub-micron Tungsten vias are examined. Process integration issues considered are the influence of oxide etch and glue layer deposition profiles on void formation during W-CVD, tradeoffs in collimated and non-collimated Ti/TiN PVD systems, and the effects of stop on oxide and stop on TiN W-etchback profiles on subsequent metallization. Simulations are tested with experimental results.

4:20 pm MS-MoA8 Reactor- and Feature-Scale Simulation of Tungsten Chemical Vapor Deposition, A. H. Labun, Digital Equipment Corp., Hudson, MA 01749.

Models of tungsten chemical vapor deposition (WCVD) by the hydrogen reduction of tungsten hexafluoride (WF₆) are presented which span both the reactor and device scales, providing the capability of predicting tungsten step coverage in features from reactor settings. Comparisons of model predictions with experiments are made at both scales. A chemical kinetic scheme consisting of 16 elementary gas-surface reactions has been adopted from the work of Arora and Pollard [1] simulating low pressure WCVD and extended to 40 Torr using SURFACE CHEMKIN software [2]. A one-dimensional, stagnation-flow model of a single wafer reactor predicts deposition rate over a wide variety of process conditions, including deviations from the desired zero-order dependence of deposition rate on WF₆ partial pressure. The 'starvation' of the final elementary deposition reaction step for intermediate adsorbed WF_x surface species, observed in the simulations, is evidently responsible for the resulting highly nonconformal deposition under these conditions. A simple model of an entire submicron feature as a single perfectly stirred reactor shows that as feature aspect ratio changes, so too does the internal chemical balance and hence the WF₆ partial pressure at which deviations from the zero-order deposition rate begin. A submicron feature in the zero-order deposition rate regime may also be modeled as a pair of communicating perfectly-stirred reactors, the first extending approximately one mean free path from the feature opening and the second comprising the remainder of the feature, with the gas concentrations at the feature opening determined by the reactor-scale simulation. The variation in starvation conditions between the two reactors which model the feature correlates with observed step coverage, from which a sticking coefficient may be deduced. The resulting sticking coefficient-based deposition model for WCVD has been integrated with a model for collimated TiN sputter deposition, permitting the submicron plug-filling process to be handled by a standard topography simulator.

[1] R. Arora and R. Pollard, J. Electrochem. Soc. 138 1523 (1991).
[2] M. Coltrin, R. Kee, F. Rupley, Sandia Report SAND90-8003B (1991).

4:40 pm MS-MoA10 Deposition and Flow Planarization of Glasses, H. Liao and T. S. Cale*, Center for Solid State Electronics Research, Arizona State University, Tempe, AZ 85287-6006.

We present our physically based simulation package (EVOLVE-

FLOW), which consists of EVOLVE [1] and a thin film thermal flow process simulation program. EVOLVE's algorithms for simulating profile evolution during low and high pressure deposition have been discussed [2]. The thermal flow process is modeled as two-dimensional, incompressible, free-boundary flow, and has been discussed by Bornside *et al.* [3]. The flow simulator generates a mesh in the deposited film, and solves the Navier-Stokes equations using the penalty function finite element method. The deposition/flow process is divided into a number of time steps, and the location of the free boundary is determined as a function of time. Within each simulation time step, the local film deposition rates are determined by EVOLVE, then the thermal flow simulation program predicts the flow induced film velocity. These velocities are combined and the free surface profile is updated using the movement algorithms in EVOLVE.

As an example application of EVOLVE-FLOW, we present simulated film profiles during the simultaneous deposition and fusion flow planarization of borophosphosilicate glasses in high aspect ratio trenches. The process is based on tetraethoxysilane-oxygen chemistry with triethylborate and phosphine as dopant sources, and is conducted in a LPCVD reactor. We compare the planarization performance of the single step process to that of a two step process (deposition then reflow) in order to explain its reported superior trench filling capability.

1. EVOLVE is a deposition process simulator developed by T. S. Cale at ASU and Motorola, Inc. with funding from the SRC and NSF.
2. T. S. Cale and G. B. Raupp, *JVST*, **B8**(6), 1242 (1990).
3. D. E. Bornside, R. A. Brown, S. Mittal and F. T. Geyling, *Appl. Phys. Lett.*, **58**(11), 1181 (1991).

BIOMATERIAL INTERFACES

Room A106 - Session BI-MoA

Cell-Solid Surface Interactions

Moderator: J. J. Hickman, Science Applications International Corporation.

2:00 pm **BI-MoA1** *The Bone Cell/Biomaterial Interface*, *J. E. Davies*, Centre for Biomaterials, University of Toronto, Ontario, M5S 1A1, Canada.

The future for Biomaterials lies in creating a marriage between materials science and biotechnology. Fundamental understanding of interfacial reactions between biological systems and artificial substrates will simultaneously lead to considerable advance in: (1) design of new materials for implantation in the body (Biomaterials), (2) creation of substrates for engineering of biological reactions *in vitro* Biotechnology), and (3) creating substrates for diagnostic tests of biological processes in health and disease (Health Care). It has long been accepted that mechanistic explanations of reactions at solid liquid interfaces, within the biological milieu, will only emerge as a result of the deconvolution of molecular events at surfaces. However, there is currently a wide gap between our understanding of the physicochemistry of surfaces and the application of this knowledge to interpretations of cell behaviour. This gap is slowly closing as a result of the considerable efforts of surface scientists who have applied themselves to biological interfacial problems, and the emergence of analytical techniques which provide information on the intact solid/liquid interface. Specific examples of the need for a surface analytical approach to understand the behaviour of both major bone cell types, osteoblast and osteoclasts, at surfaces will be provided. These examples will include studies of corrosion behaviour, as a function of passivation treatment, of metal alloys in biological systems; bone cell adhesion and cell spreading as functions of the charged, or polar, nature of a polymer substrate; changes in bone protein adsorption as a function of polymer surface modifications and the means by which bone growth can be dictated by materials surfaces.

2:40 pm **BI-MoA3** *Human Neutrophil Motility on Modified Surfaces*, *L. Harvath, N. E. Brownson, K. E. Foster¹, J. J. Hickman¹, CBER FDA, Bethesda, MD 20892, ¹SAIC, McLean, VA 22102.*

Human neutrophil migration in the absence of a stimulus (random motility) and in the presence of various concentrations of a chemotactic stimulus (chemotaxis) was evaluated on modified and unmodified polycarbonate membranes. Neutrophils are the first blood cells found at an inflammatory site. The assay is routinely used to evaluate neu-

rophil migration through 5 μ m pores in the polycarbonate membranes in response to medium alone or the chemoattractant, N-formylmethionyl-leucyl-phenylalanine (FMLP). Neutrophils that have migrated from the upper surface through the pores to the lower polycarbonate membrane surface are quantified by image analysis (Harvath *et al.*, 1980, *J. Immunol. Methods*, **37**: 39-45). To investigate the role of the surface in neutrophil responses to a chemotactic agent, we modified the polycarbonate membranes with various self-assembled monolayers (SAMs). The silane monolayers used in this study contained various functionalities, including amines, thiols, and hydrophobic groups. The surfaces were analyzed by X-ray photoelectron spectroscopy and contact angle measurements. These modifications increased neutrophil random migration, in some cases, 3 to 4-fold over control responses. The surface modifications did not inhibit neutrophil chemotactic responses to FMLP, however, amine-containing modifications substantially increased (>6-fold) the chemotactic response to suboptimal concentrations of FMLP. The significance of enhanced neutrophil migration to suboptimal concentrations of chemoattractant may be directly relevant to *in vivo* responses to implanted materials.

3:00 pm **BI-MoA4** *Nanofabricated Structures for the Measurement of Signals from Single Cells*, *J. M. Cooper, A. Griffiths, and H. Morgan*, Bioelectronics Group, Department of Electronics, University of Glasgow, Glasgow, G12 8LT, UK.

Dielectrophoresis is a technique which enables non-uniform electric fields generated at micro-electrodes to be used in order to guide or to position cells on the basis of their dielectric properties. By fabricating suitable geometries of such electrodes, it has proved possible to position individual cells within a two dimensional array of nano-scale amperometric biosensors. The sensors, which are based upon the immobilisation of cytochrome c on self-assembled thiol monolayers on gold, have a potential application in the detection of superoxide for cell signalling. To this end, measurements have been made on stimulated single neutrophils.

3:20 pm **BI-MoA5** *Cell-Solid Surface Interactions*, *D. A. Okrongo*, Xytronyx, Inc., San Diego, CA 92121.

Methods to effect specific cell adhesion to biomaterials is an area of intense investigation. In addition, recombinant protein technology has created a number of new biological drugs for diagnosing and treating disease. Combination of the products of biotechnology with biomaterials research has created an exciting new field of hybrid medical devices. A hybrid medical device can be defined as any medical device that uses a biological material, usually a recombinant protein, to effect its intended medical application. The current investigation will be a case study of a medical device used in human clinical trials to separate immune stem cells and CD8+ lymphocytes. The discussion of the methodology employed in the development of these highly specific cell adhesion devices will highlight a new type of biomaterial, surface analysis techniques such as ESCA and Attenuated Total Reflectance FT-IR and the use of monoclonal antibodies directed to cell surface antigens. Preliminary results of human clinical trials will be presented.

INVITED

4:00 pm **BI-MoA7** *Nanofabricated Fibers for Studying the Phagocytosis of Inorganic Particulates by Macrophages*, *J. Gold, B. Nilsson, and B. Kasemo*, Chalmers University of Technology and University of Göteborg, 412 96 Göteborg, Sweden.

Inorganic particulates occur in the body due to e.g. wear debris generated by articulating surfaces in orthopaedic implant devices, or by inhalation of mineral dusts and fibers. Empirical observations have indicated that these particulates can lead to failure of the implant and lung diseases, respectively. Macrophages attempt to remove particulates from the body via phagocytosis into the cell, and send out signals indicating the presence of either an inert or a toxic foreign object. Several physical and chemical characteristics of the particulates appear to play a role in determining this phagocytic response of macrophages.

Typically, particulates used in these types of studies are produced by e.g. grinding raw/bulk materials, spin casting, or atomization. However, these techniques generate inherent distributions in the size, shape and chemical composition of particulates even within a given production lot. In the present work we have produced fibers having controlled shapes, dimensions, sized distributions, and compositions for studying the role of these factors in the cellular response. By using nanofabrication techniques, fibers can be made in a large number of materials and in a wide range of dimensions (nm's to mm's). Significant quantities for cell culture studies can be made with tight control over the fiber size distribution.

Ti, SiO₂, and Au fibers of dimensions 0.1, 1 and 10 μ m, have been

S. Estes, M. J. Fleming, R. Gaylord, C. Gow, W. Syverson, IBM Microelectronic Division, Essex Junction, VT.

The combination of an HF etch and RCA clean is widely recognized as an effective cleaning process to remove metallic and particulate contamination from silicon wafers. Recently it has been observed that these cleaning processes may also cause nanoscale roughening of silicon surfaces and generate silicon pillars (spikes) and light point defects (LPDs), both potential yield detractors.

Atomic Force Microscopy (AFM) was used to measure these silicon features as a function of etching variables. This paper will discuss the role of process chemistry (temperature, pH, megasonics, time) and metallic contamination on pillar formation. Interestingly, in contrast to previous literature, pillar formation has been observed on both hydrophilic and hydrophobic wafers. Comparison of AFM features with conventional light scattering data will also be shown.

In addition to conventional post clean analysis data, we will also show in-situ AFM results which allow one to directly observe changes in surface morphology during a simulated cleaning process.

11:00 am EMMS-TuM9 Low Damage Surface Cleaning of CdTe by Hydrogen ECR Plasma, Yi Luo, Peter Lasky, Ming Chang Shih, and R. M. Osgood, Jr., Microelectronics Sciences Laboratories, Columbia University, New York, NY 10027.

We present a low damage, in-situ process for surface cleaning of CdTe semiconductor by hydrogen Electron Cyclotron Resonance (ECR) plasma. The hydrogen ECR plasma is generated by a 2.5 GHz microwave source operated at 25-30 W. The pressure in the ECR processing chamber is about 5×10^{-4} Torr. Surface cleaning of CdTe by Ar-ion sputtering has been reported, however it is known that the ion bombardment can cause surface damage. By using reactive hydrogen atoms generated in an ECR plasma with low kinetic energy to remove surface oxides, surface damage can be avoided. CdTe (110) single crystal surfaces were used in our experiments. X-ray photoelectron spectroscopy (XPS) and Auger spectroscopy are used to trace the change of the surface composition due to hydrogen ECR plasma processing. Low energy electron diffraction (LEED) is used to monitor the surface structure after cleaning. The Te(3d), Cd(MNN) and O(1s) XPS spectra show that at room temperature, a hydrogen ECR plasma can remove CdTe surface oxides effectively. We also present the results of the oxide reduction rate at various substrate temperatures. In addition, hydrogen ECR surface cleaning for both a native oxide and an oxide grown by oxygen ECR is discussed. The results of surface cleaning by hydrogen ECR are compared with those of Ar-ion sputtering and thermal desorption methods. Our results show that CdTe surface oxide can be grown and removed effectively by using ECR plasmas. This work is supported by National Science Foundation.

11:20 am EMMS-TuM10 The Thermal Stability and Effect of Atomic Deuterium Exposure on S-Passivated InP(100)-(1 x 1), G. W. Anderson, M. C. Hanf and P. R. Norton, University of Western Ontario, London, Ontario, Canada and Z. H. Lu and M. J. Graham, National Research Council, Ottawa, Ontario, Canada.

Surface cleaning/passivation is a key step in semiconductor manufacturing processes, such as epitaxial overgrowth. For InP(100), the conventional approach has been to remove the native oxide by annealing at 820K. This technique cannot be utilized in some situations (i.e. growth on a patterned surface) where the thermal budget is very low, as such high temperatures would destroy the initial structure. In this investigation we have examined the potential of S passivation in an aqueous $(\text{NH}_4)_2\text{S}$ solution as a low temperature technique to produce InP(100) substrates suitable for further processing.

We have investigated the thermal stability and effect of atomic deuterium exposure on S-passivated InP(100) - (1 x 1). The S passivated surfaces are found to be thermally stable to ~700K where the samples begin to evaporate, giving rise to roughened surfaces displaying macroscopic In islands. Atomic deuterium exposure was observed to result in the removal of S from the surface. At elevated temperatures (>500K) a clean InP(100) - (4 x 2) reconstructed surface is formed, which shows a regular distribution of In islands. For samples held at 300 or 400K a D-terminated InP(100) - (1 x 1) surface is obtained, which shows the same morphology as the initial sample.

These results illustrate several potential methods for producing InP(100) substrates suitable for use in semiconductor manufacturing. Depending on the substrate requirements, S-passivation can be utilized to produce a S-terminated (1 x 1), D-terminated (1 x 1) or clean (4 x 2) InP(100) surface at low temperature.

11:40 am EMMS-TuM11 Thermal Effects on GaAs(001) Surface Prepared by Deoxygenated and Deionized Water Treatment, Y. Hirota, T. Ogino, *Y. Watanabe, and *M. Oshima, NTT Basic Research Labs, 3-1, Morinosato Wakamiya, Atsugi-shi, Kanagawa 243-01, Japan. *NTT Interdisciplinary Research Labs, 3-9-11 Midori-cho, Musashino-shi, Tokyo 180, Japan.

Surface defects of GaAs degrade optical and electrical device characteristics such as efficiency of luminescence intensity and electron mobility. To prevent surface defects during cleaning process, we have recently proposed a new method based on electrochemical reaction. Deoxygenated and deionized water (DODIW) treatment, in extremely low concentration of dissolved oxygen (1 ppb), completely removes surface oxides and produces a passivation layer of elemental As and/or hydrogenated As on the surface due to the liberation of Ga atoms after removal of oxides.¹ After heating the DODIW treated-GaAs (001) and (111)B surfaces in high vacuum, RHEED observation shows spotty 2 x 4 and 3 x 6 surface reconstruction patterns,² and STM observation shows 2 x 2 and $\sqrt{19} \times \sqrt{19}$ structures, respectively.³

In the present study, we investigate the temperature dependence of surface Fermi level (E_F) by using synchrotron-radiation photoelectron spectroscopy (SRPES) in ultra-high vacuum. SRPES reveals that the E_F for the DODIW treated n-type ($8 \times 10^{16}/\text{cm}^2$) GaAs (001) surface lies at $E_{\text{VBM}} + 0.9$ eV at room temperature, moves toward $E_{\text{VBM}} + 0.6$ eV on heating to 500°C, and returns to near-flat-band position ($E_{\text{VBM}} + 1.0$ eV) after cooling to room temperature. These results suggest that the changes in E_F is due to the reduction in density of surface state, which is achieved with the DODIW treatment. This is because the DODIW treatment is essentially a low energy process and the DODIW treated surface is initially passivated with excess elemental As and/or hydrogenated As layer.

¹Y. Hirota, *J. Appl. Phys.*, 75 (1994) 1798.

²Y. Hirota, Y. Homma, and K. Sugii, *Sur. Sci.* 60/61 (1992) 619.

³T. Fukuda, and Y. Hirota, *J. Vac. Sci. Technol.* B11 (1993) 1982.

MANUFACTURING SCIENCE AND TECHNOLOGY

Room A110 – Session MS-TuM

Manufacturing Overview and Environmental Issues

Moderator: G. W. Rubloff, North Carolina State University.

8:20 am MS-TuM1 Environmental Consciousness: A Strategic Competitiveness Issue for the Microelectronics Industry, Gregory E. Pitts, Director, Environmental Programs, Microelectronics and Computer Technology Corp. (MCC), 3500 W. Balcones Center Dr., Austin, TX 78759-5398, (512) 338-3790, (512) 338-3814, pitts@mcc.com.

The electronics industry has become increasingly aware of the need to develop a strategic approach to identify cost-effective, long-term solutions for environmental issues. Many organizations have invested resources to address environmental issues and business opportunities. As these efforts have evolved, it has become apparent that a strategic, coordinated approach for addressing environmental issues and business opportunities will help the electronics industries maintain a competitive edge in the international market and keep up with competitors who have long-range planning partnerships in the industry and government for addressing environmental issues. This paper will discuss several activities to develop a broad environmental strategy and solutions for priority environmental issues.

INVITED

9:00 am MS-TuM3 Integrating Regulatory Policy & Science—Can It Be Done? Bruce C. Jordon, Director, Emission Standards Division, MD-13, U.S. EPA, Research Triangle Park, NC 27711.

Regulatory activity within the United States is placing a heavy burden on industry. The economic consequences of government regulations have increased many fold in the last few years. Too often, industry has left in the hands of lawyers, and non-scientific oriented employees, or professional lobbyist its role in regulatory activity. Yet, within most industries lies the knowledge and capability to achieve the most cost-effective approach to meeting environmental objectives.

This presentation will review how industry and the academic community can take a more active role in regulation development. A review of the regulatory process for the Clean Air Act will be presented.

Examples where industry can and have become actively involved in regulation development will be discussed and results reviewed. Finally, the future of the relationship between the industry and regulator is outlined and options illustrated.

9:40 am MS-TuM5 Environment, Safety and Health Issues in Manufacturing.

One of the roadmaps in the National Technology Roadmap for semiconductor technology is the 15 year strategy for Environment, Safety and Health improvements in semiconductor chips and package manufacturing. Materials and process changes are a significant part of this strategy which will be discussed in this paper.

SEMATECH, a consortium of leading United States semiconductor manufacturers and ARPA, is a major implementor/facilitator of the national roadmap. The SEMATECH ESH program trends in semiconductor manufacturing and their relation to the national roadmap will be presented.

INVITED

10:20 am MS-TuM7 Factory of the Future: The "Whole Factory" View, Dr. Sam Harrell, Chief Strategy Officer, SEMATECH.

The primary task of a semiconductor factory is to produce integrated circuits of outstanding performance at a steeply declining cost per electronic function delivered. The semiconductor industry over the next decade will need to continue to drive costs down in order to maximize manufacturing productivity in the climate of rising product complexity, equipment cost, and risk. Historically we have concentrated on aggressive yield improvement and declining wafer fab cost per cm² as the dominant factors for increasing productivity. To continue to be competitive, SEMATECH is extending its focus beyond cost per cm² to the more total view of cost per function. We are working toward a combination of solutions to meet our manufacturing challenges recognizing that no single solution will provide enough benefit to achieve the productivity gains end users expect. The "Whole Factory" View is a useful strategic planning tool for defining the future of manufacturing in the semiconductor industry.

INVITED

11:00 am MS-TuM9 Research Opportunities in Semiconductor Manufacturing Science and Technology, Daniel J. C. Herr, The Semiconductor Research Corporation, 79 Alexander Drive, Research Triangle Park, North Carolina 27709.

This talk describes several opportunities for long-range research at the academic-industrial interface that could significantly impact future generations of semiconductor manufacturing technology. A vision for success is emerging within the semiconductor industry that calls for increased cooperation and collaboration in tackling critical precompetitive issues. This vision is driven by the need for leading-edge, high-performance, semiconductor-based electronics. It comprehends the importance of balancing the escalating cost, complexity, and sophistication of the research and development processes required to bring new science and technology to an environmentally conscious market place. The pressure to continuously shorten product and process research and development cycle times is engendering effective and innovative working partnerships between independent companies, government, and university R&D programs. These pioneering efforts have led to industry consensus on identifying several critical gaps and potential showstoppers on the path to future technologies. These recognized critical issues provide opportunities for long-term research that will increase fundamental understanding of chemical and physical processes, enable high-performance and robust technologies, and strengthen the supporting infrastructure of trained scientists and engineers.

INVITED

11:40 am MS-TuM11 Photocatalytic Oxidation for Point-of-Use VOC Abatement in Microelectronics Manufacturing, Mahbub Ameen, Ronnie Varghese, Jill Nico, and Gregory B. Raupp, Department of Chemical, Bio & Materials Engineering, Arizona State University, Tempe, AZ 85287-6006.

In response to the Montreal Protocol of 1989, many microelectronics manufacturing firms are replacing chlorofluorohydrocarbons (CFCs) used in cleaning processes with volatile organic compounds (VOCs) such as acetone, isopropanol and glycol ethers. Although these solvents provide acceptable cleaning capabilities, these, as well as other VOCs, are on the 1990 Clean Air Act Amendments list of hazardous compounds. As a result, a threshold has been established for VOC emissions, forcing the industry to consider VOC abatement equipment to limit their emissions. We have recently initiated a research and development program aimed at establishing the commercial viability of gas-solid heterogeneous photocatalytic oxidation (PCO) for point-of-use abatement of VOCs in air streams. In PCO, VOCs present in

process or air vents can be rapidly and completely oxidized to innocuous byproducts over near-UV illuminated titanium dioxide thin film catalyst at room temperature. Photocatalytic oxidation appears to be well-suited to the special requirements of the semiconductor processing industry. In this paper we review these requirements in the context of the recently-published SIA roadmap. The specific requirements for VOC abatement from (1) a typical photolithography track, and (2) solvent cleaning stations are presented, as are bench scale PCO kinetics for target VOCs.

BIOMATERIAL INTERFACES

Room A106 – Session BI-TuM

Protein-Solid Surface Interactions

Moderator: B. D. Ratner, University of Washington.

8:20 am BI-TuM1 Molecular Recognition at Protein-Biological Composite Interfaces, P. S. Stayton, R. Clark, C. L. Long, L. Klumb, A. Chilkoti, A. A. Campbell, G. Drobny, University of Washington, Seattle, WA 98195 and Battelle Pacific Northwest Laboratories, Richland, WA 99352.

The Materials Science community has focused much recent attention on the way in which biological composites and ceramics are synthesized and processed. Biology provides many examples of exquisitely constructed hard materials, ranging from bone to seashell nacre, and "lessons from nature" may prove valuable in attempts to improve ceramic/composite processing strategies. In most known examples, proteins provide the molecular control necessary to control the hierarchical microstructure of composites with unparalleled specificity and structural resolution. Despite their well established importance, however, there is little known of the direct molecular recognition mechanisms used by protein surfaces to control the nucleation and growth of biological composites such as calcium oxalate and hydroxyapatite. We have utilized a combination of site-directed mutagenesis, functional characterization of protein-crystal interactions, and high-resolution structural analysis to elucidate molecular recognition processes at the protein-biological composite interface. These studies are providing a detailed molecular picture of the mechanisms utilized by proteins to control composite structure. The principles and rules defining biological composite engineering should be generally useful to materials scientists interested in improving ceramic engineering technologies.

INVITED

9:00 am BI-TuM3 Surface Plasmon Imaging of Biotin-Streptavidin Binding on UV-Photopatterned Alkanethiol Monolayers Self-Assembled on Gold, D. Piscevic, M. Tarlov*, and W. Knoll, Max-Planck Institute for Polymer Research, Ackermannweg 10, D-6500 Mainz, Germany and *National Institute of Standards and Technology, Gaithersburg, MD 20899.

We report the surface plasmon imaging of UV-photopatterned alkanethiol self-assembled monolayers (SAMs) on gold and subsequent biomolecular recognition reactions on these surfaces. To photopattern the monolayers, a SAM of desired surface functionality is formed, irradiated with UV light through a mask, and then immersed in a solution containing a second alkanethiol molecule of different terminal functionality. Using this procedure, patterned monolayers are formed containing two regions of different surface properties with micron-scale features. Surface plasmon resonance was used to ascertain the effectiveness of the UV-photopatterning process by measuring changes in SAM thickness following UV exposure and immersion in a second alkanethiol solution. The binding of the protein streptavidin to biotin-terminated alkanethiols was used as the model biomolecular recognition system. The specific binding of streptavidin to the surface was maximized by using a mixed monolayer consisting of the biotinylated thiol diluted into a hydroxy-terminated SAM. A pure hydroxy-terminated monolayer was used to retard the non-specific binding of streptavidin to selected regions of the surface. In this talk the use of surface plasmon microscopy to detect and image in real time the binding of streptavidin to the patterned biotinylated SAM surfaces will be demonstrated. In addition, the potential use of surface plasmon microscopy for parallel addressing of biosensing and diagnostic arrays will be discussed.

beam propagation method. The loss for curved waveguides increases drastically for radii of curvature shorter than 400 μm , which is in good agreement with experiment results from a simple test chip with integrated LEDs, Si-mirrors, curved waveguides and LED-based detectors.

In summary, the fundamental technologies to realize the optoelectronic integrated circuits have been developed. INVITED

2:40 pm EM-TuA3 Silicon Optical Bench Waveguide Technology, *Charles H. Henry*, AT&T Bell Laboratories, Murray Hill, N.J. 07974.

This technology makes optical waveguides similar to those in optical fibers by depositing thick films of doped silica on silicon substrates, primarily by low pressure chemical vapor deposition. The films are patterned by photolithography and etching into complex optical integrated circuits. Components include taps, splitters, star couplers, broad and narrow band multiplexers and low speed switches. Commercial application of optical integrated circuits require nearly perfect optical waveguides with very low loss, polarization independence, control of the absolute refractive index and control over the optical phase difference between different paths. The SiOB waveguide technology has the potential to meet all of these needs. The waveguide fabrication, properties, components of current interest and opportunities for systems applications areas will be reviewed. INVITED

3:20 pm EM-TuA5 Novel LSI Memories with Optical Interconnections, *M. Koyanagi*, Tohoku University, Sendai, Japan.

The performance of the microprocessor has been significantly improved owing to the progress of LSI technology. However, even if the microprocessor performance is rapidly improved, the overall performance of the computer system is not always so rapidly improved because it is eventually limited by the data transfer speed or the data bandwidth of the buses. In particular, this bottleneck in the bus becomes more serious in the parallel processing computer system. We have been interested in employing optical interconnection as an intrachip or interchip interconnection to increase the data transfer speed among processors and memories in the parallel processing computer system. In this paper, we describe new LSI memories with optical interconnection such as Optical RAMbus (ORAMbus) memory. Optical RAM (ORAM)-link and Optically Coupled Common (OCC) memory which have been proposed to increase the data transfer speed and the data bandwidth by transferring data among many memories through optical interconnection. These memories have three-dimensional (3D) structure which consist of several 2D memory layers with LEDs and photoconductors or photodiodes. The memory layers are connected each other through many free-space optical interconnections in the vertical direction. The basic functions and performances of these new memories are evaluated using an optical-electrical circuit simulator. In addition, the test chips are fabricated to confirm their basic functions. INVITED

4:00 pm EM-TuA7 Rare Earth Doped Silicon Emitters, *J. Michel and L. C. Kimerling*, Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, MA 02139.

Recent results on the improvement and understanding of light emission from the Si:Er system are reviewed. Erbium emits light at 1.54 μm , a wavelength ideal for optical communication. While most recent studies focused on co-doping with oxygen to optically activate erbium, fluorine co-doped samples show two orders of magnitude efficiency improvement at low temperatures. Room temperature performance of Si:Er based edge emitting LED's, capable of direct waveguide coupling, will be shown and discussed in the context of system requirements for integrated optical interconnection. INVITED

4:40 pm EM-TuA9 SiGe/Si Quantum Well Light Emitters, *Yasuhiro Shiraki and Susumu Fukatsu*, Research Center for Advanced Science and Technology, The University of Tokyo, Komaba, Meguro-ku, Tokyo 153, Japan.

Highly luminescent SiGe/Si heterostructures are now fabricated by various growth techniques such as molecular beam epitaxy (MBE), UHV/CVD, rapid thermal CVD and so on. Especially, gas source MBE where hydrogenated gases, Si_2H_6 and GeH_4 , are used as molecular beam sources can provide extremely promising results for light emitting device application. Quantum wells (QWs) grown by this method show a clear quantum confinement effect as the well width is changed and their luminescence properties are well understood in terms of effective mass approximation. The temperature dependence reflects the band alignment at the heterostructures and carrier dynamics in multi-QWs provide strategies to realize light emitting devices. These results indicate that device properties of this system are predictable

from the quantum mechanical consideration, suggesting high potentials in band engineering and device designing. Some examples of LEDs including room temperature operation are demonstrated and new concepts of optical devices exploiting the nature of this material system are proposed.

INVITED

MANUFACTURING SCIENCE AND TECHNOLOGY
Room A110 - Session MS-TuA

Advanced Manufacturing Equipment - A

Moderator: *M. Liehr*, IBM TJ Watson Research Center.

2:00 pm MS-TuA1 The Semiconductor Equipment Industry: Applied Materials Role and Growth, *Robert Z. Bachrach*, Applied Materials, 3050 Bowers Ave., MS 1250, Santa Clara, CA 95054.

Applied Materials celebrated its 25th anniversary in 1993 and in 1994 became the first Semiconductor Equipment Manufacturer to achieve revenue of one billion dollars per year. An overview will be presented of some of the factors and circumstances that have allowed Applied Materials to differentiate itself in the industry both technically and in business performance. Applied Materials now provides its customers with advanced manufacturing equipment for Etch, CVD, PVD, Implant, and HTF (High Temperature Films) for 6" / 8" wafer processing. Applied Materials has focused on single wafer processing with common architecture multi-chamber mainframes: Precision 5000[®], Endura[®], and CenturaTM. A major thrust is to extend these capabilities to the next wafer size of 12" and 14" silicon which is expected to be manufacturing starting in 1998. INVITED

2:40 pm MS-TuA3 Advances in Semiconductor Manufacturing Equipment, *Evert van de Ven, Tom Bowman*, Novellus Systems, San Jose, California.

The Semiconductor Industry is maturing rapidly and with its requirements for manufacturing equipment have been changing from process capable hardware to turnkey production systems with guaranteed performance.

This paper will review the main issues critical to high volume manufacturing and how changes, historically and in the future, are affecting the equipment requirements and design approach. With these changes, the main focus on equipment is shifting to productivity cost/ performance and production reproducibility. To determine performance to these requirements, new metrics, such as equipment productivity index, value added time, and sigma six processing are being added to defect density and cost of ownership. Using these and other criteria, the advantages and limitations of various equipment concepts will be compared and examples of successful implementation presented. Finally, an overview will be given of the challenges for next generation equipment. INVITED

3:20 pm MS-TuA5 MESC Cluster Tools for Advanced Metalization, *P. H. Ballantine, T. Omstead, M. Moslehi*, CVC Products, Inc., Rochester, NY 14603.

Cluster tools are playing an increasingly important role in semiconductor manufacturing. Individual process steps may be integrated into a single vacuum environment to reduce surface contamination and minimize operator handling. By using a modular approach to system design, individual process modules may be taken off line for service and burn in while the rest of the tool remains in production, thus reducing qualification time. From an equipment development point of view, process modules may be developed and characterized independent of the wafer transport platform and cluster control architecture. This provides a significant reduction in time-to-market for new process equipment. The adoption of the SEMI MESC (Modular Equipment Standards Committee) standards has provided additional benefits by allowing two or more equipment suppliers to integrate "best of breed" process modules into a single cluster tool. We have used our CVC Connexion platform to rapidly develop thin film deposition tools for both the data storage and semiconductor industries. Process modules for PVD, RTP, MOCVD, and soft etch have been developed in parallel and combined for use in production of thin film recording heads and development of advanced multilevel metalization schemes. Portions of the process modules have been based on tech-

nology licensed from the MMST program at Texas Instruments. We will present examples of individual process results as well as calculations of cost of ownership for specific integrated applications.

3:40 pm MS-TuA6 Application of Motorola IRONMAN Methodology to Equipment Reliability Improvement, Robert Duffin, Kathleen McCormack, Motorola SPS.

The cost to build and equip a modern IC manufacturing area is increasing rapidly. At the same time the useful life of the area is being shortened by the decreasing time required to develop new IC products. Some trend watchers have even questioned the economic viability of the IC industry past the 64 megabit DRAM.

These trends will be countered with faster fab startups, a better ROI early on, and fab life extensions. Improvements such as these will require manufacturing equipment to achieve a high degree of reliability immediately after installation and have the potential to be upgraded in the field for next generation products. These upgrades must be fully qualified at the equipment supplier site before commercial introduction.

There is a clear need for equipment suppliers and IC manufacturers to work together in a systematic way to achieve these goals. This will require an equipment development and improvement methodology that gets accurate information to the equipment manufacturer quickly to accelerate cycles of learning. Reliance on field data will slow this down and may even be misleading. If equipment manufacturers can develop and evaluate new equipment and processes in their factories, accuracy and speed will increase and the time required to improve reliability will be greatly reduced.

Motorola has developed, and successfully used, a methodology called IRONMAN for working with equipment suppliers to achieve mutual reliability and speed-of-introduction goals. This paper gives specific examples of how IRONMAN works and proposes an industry wide application of the principles involved.

4:00 pm MS-TuA7 The Development of the Fast Thermal Processor (FTP), Katsuya Okumura, Toshiba.

Furnace operations (oxidations, anneals and LPCVD) account for a large percentage of integrated circuit fabrication cycle time and short cycle time is necessary to minimize product development time and manufacturing capital investment. Therefore, further innovation is needed in the areas of furnace tooling and processing in order to improve ULSI fab productivity.

Today, hot processes are carried out in either conventional furnaces or newer rapid thermal processors (RTPs). Furnaces have relatively slow ramp rates (3–10°C/minute) compared to RTPs (500–1000°C/minute), but furnaces can typically process 100–150 wafers at once while RTPs are usually single-slice (single-wafer) processors. Also, there exists a large body knowledge related to conventional furnaces and processes which has yet to be duplicated in RTPs. But because of the large difference in operating regimes, it is difficult to transfer learning between the two types of equipment. Moreover, there are many unresolved problems that are unique to RTP.

In order to combine the best features of furnaces and RTPs, Toshiba and TEL collaborated to develop the Fast Thermal Processor (FTP). The FTP utilizes a standard vertical furnace configuration with a small batch size (50 wafers) and enhanced heating and cooling capability to achieve ramp rates of 50–100°C/minute. In addition, thermal and CVD processes can be done sequentially in the same chamber to achieve cost-effective process development and manufacturing. **INVITED**

4:40 pm MS-TuA9 Low Thermal Budget Gap Filling for Semiconductor Manufacturing, Kevin J. Uram, John K. Shugrue, and Nathan P. Sandler, Lam Research Corp., 49026 Milmont Dr., Fremont, CA 94538.

Advanced device designs and new material compatibility are placing greater demands on the gap filling capability of interlayer dielectric films. In this paper we present results of recent studies of the deposition of borophosphosilicate glass (BPSG) from the reaction of tetraethylorthosilicate (TEOS), triethylborate (TEB), phosphine (PH₃), and oxygen in the temperature range of 750°–800°C for high aspect ratio gap filling and interlayer isolation applications. Our studies have shown that at least three different mechanisms contribute to gap filling by BPSG in this temperature range. Viscous flow of the deposited glass is the predominant mechanism for gap fill at temperatures in excess of the glass transition temperature. A simple physical attraction between approaching side-walls has been observed to contribute to gap fill. Finally, some degree of chemical selectivity can be achieved from the deposition of BPSG from the above mentioned reactants. This chemical selectivity contributes to preferential gap fill from the corner

of structures. All of these mechanisms contribute to gap filling capability of BPSG on high aspect ratio structures (>2.5:1) with small openings <0.17 μm at temperatures as low as 750°C.

5:00 pm MS-TuA10 Sub-Atmospheric CVD (SACVD) Ozone/TEOS for SiO₂ Trench Filling, I. Shareef¹, G. W. Rubloff², M. Anderle¹, W. Cill³, J. Cotte¹, and D.-H. Kim²; ¹IBM Research, Yorktown Heights, NY 10598, ²North Carolina State Univ., Raleigh, NC 27695, ³Rensselaer Polytechnic Institute, Troy, NY.

Equipment for *sub-atmospheric CVD (SACVD)* is already in commercial use for ozone/TEOS processes to achieve low temperature (400°C), conformal, planar SiO₂ films for interlevel dielectric and possibly trench isolation applications, despite experimental complexities and poor understanding of its multicomponent gas and surface reactions. We report the first systematic study kinetics, conformality, and material properties over a broad pressure range (30–300 Torr), using an ultraclean MOCVD reactor with showerhead, O₃ generator, liquid TEOS delivery system, and O₃ quantification by chemical reaction/titration. SACVD rates exceed those for atmospheric pressure CVD; rates increase and then saturate with either O₃ or TEOS overabundance, providing an intermediate process window for optimizing rates, conformality, and film quality. Increasing O₃/TEOS ratio decreases rates but enhances SiO₂ quality (O₃/TEOS > 3 is required for low BHF etch rates, indicative of good SiO₂ quality). Conformality and trench filling properties at 30, 200, and 280 Torr are enhanced for higher O₃/TEOS ratio and higher total pressure, as seen for shallow and deep trenches (aspect ratios ~1.5 and 10, respectively), with excellent conformality observed for shallow trenches. Gas phase deposition precursors and O₃ supply appear crucial in assuring complete oxidation of film constituents and volatilization of organic products.

BIOMATERIAL INTERFACES
Room A106 – Session BI-TuA

The Biosensor-Biology Interface

Moderator: J.-J. Pireaux, Universitaires Notre-Dame, Belgium.

2:00 pm BI-TuA1 In Vivo Electrochemical Sensors: The Challenge of Achieving Biocompatible Devices, M. E. Meyerhoff and C. Espadas-Torre, Department of Chemistry, The University of Michigan, Ann Arbor, MI 48109.

The effective management of critically ill patients usually requires the frequent measurement of blood gases (pH, PCO₂, and PO₂) and electrolytes (Na⁺, K⁺, Ca⁺⁺, and Cl⁻) in whole blood. In recent years, there has been an increase in efforts to provide such test results at the patient's bedside on a continuous basis via the use of intra-arterial chemical sensors. Such *in vivo* sensors can be based on either electrochemical or optical measurements. The electrochemical type catheters are essentially miniaturized versions of the polymer membrane-based sensors already in use within commercial *in vitro* blood gas-electrolyte instruments. The reliability of values obtained by these small devices within the *in vivo* environment depends on chemical and instrument stability (drift), as well as sensor biocompatibility (thrombus formation). Indeed, adhesion of cells to the surface of implanted sensors will cause significant errors in blood gas/pH measurements owing to local changes in these values due to the cellular metabolic activity present at the sensor/blood interface. Reducing thrombus formation represents an especially difficult challenge with respect to the polymer materials used for fabrication/coating of the sensors, since chemical derivatization of the materials used (e.g., immobilization of heparin, etc.) can dramatically alter the analytical response properties (including selectivity) of the catheters. Approaches aimed at overcoming this biocompatibility issue with respect to developing implantable potentiometric ion-selective sensors will be the main focus of this paper. Specifically, the modification of polymer membrane ion-selective electrode materials (various polyurethanes) via cross-linked polyethylene oxide hydrogels, etc., will be shown to reduce platelet adhesion while maintaining electroanalytical performance of the electrochemical devices.

INVITED

2:40 pm BI-TuA3 Neural Microsensors for Automated Toxicity and Pharmacology Assays, D. A. Stenger¹, V. C. Kowtha, P. P. Bey, Jr., D. Borkholder¹, G. T. Kovacs¹, K. E. Foster², and J. J. Hickman²,

terials Research Laboratory, University of Illinois, 1101 West Springfield Avenue, Urbana, IL 61801.

Epitaxial metastable zincblende-structure β -GaN(001) 4×1 has been grown on MgO(1 \times 1) by reactive-ion molecular beam epitaxy in which the primary source of nitrogen is a low-energy (35 eV) N_2^+ ion beam extracted from an ultra-high vacuum hot-cathode single-grid source with magnetic confinement. The incident N_2^+ to thermal Ga source ratio at the substrate was 2.0 with a film growth temperature of 650°C. Film thickness range from 1 to 1.5 μ m. The temperature-dependent (10–300 K) optical bandgap $E_g(T)$ of epitaxial metastable zincblende-structure β -GaN(001) 4×1 has been determined by modulated photoreflectance and used to interpret low-temperature photoluminescence spectra. E_g in β -GaN varied from 3.302 ± 0.004 eV at 10 K to 3.231 ± 0.008 eV at 300 K with the temperature dependence given by $E_g(T) = 3.3022 - 6.697 \times 10^{-4} T^2/(T + 600)$ eV. The spin-orbit splitting Δ_0 in the valence band was determined to be 17 ± 1 meV. The oscillations in the photoreflectance spectra were very sharp with a broadening parameter Γ of only 10 meV at 10 K. Γ is comparable to modulated electoreflectance data from GaAs obtained at 4.2 K and, hence, an indication of the relatively high quality of the epitaxial β -GaN layers. The dominant transition observed in temperature-dependent luminescence was attributed to radiative recombination between a shallow donor, at ≈ 11 meV below the conduction band edge, and the valence band.

*Present address: Solar Cells Inc., 1702 N. Westwood Ave., Toledo, Ohio 43607.

EM-TuP22 Low Pressure Metalorganic Chemical Vapor Deposition of GaN on c-plane (0001) Sapphire Substrates, C.-Y. Hwang, W. E. Mayo, Department of Mechanics and Materials Science, Y. Lu, Department of Electrical and Computer Engineering, Rutgers University, Piscataway, NJ 08855, and H. Liu, Encore Corp., Somerset, NJ 08873.

GaN films were grown on c-plane sapphire substrates by low pressure metalorganic chemical vapor deposition (LPMOCVD) using a GaN buffer layer grown at lower temperatures. The quality and surface morphology of the GaN films were strongly affected by the growth conditions of the buffer layer as well as those of the GaN film. We have achieved a carrier concentration in the range of $2 \times 10^{16} - 5 \times 10^{17}$ and mobility in the range of 100–300 $\text{cm}^2/\text{V}\cdot\text{s}$. A systematic study of the influence of the buffer layer to the GaN film properties will be reported including the effects of the buffer layer thickness, growth temperature and the annealing of the buffer layer. The GaN films were evaluated by photoluminescence, Hall measurement, X-ray rocking curve, transmission and scanning electron microscopy.

EM-TuP23 Growth of Crystalline 3C-SiC Films on 6H-SiC at 900°C by Chemical Vapor Deposition from Silacyclobutane, C. Yuan and A. J. Steckl, Nanoelectronics Laboratory, Department of Electrical and Computer Engineering, University of Cincinnati, Cincinnati, OH 45221-0030; J. Chaudhuri and R. Thokala, Department of Mechanical Engineering, Wichita State University, Wichita, KS 67260-0035; M. J. Loboda, Dow Corning Corporation, Midland, MI 48686-0994.

Reduction of the growth temperature has been an important goal in the fabrication of SiC devices. We have previously reported the successful growth of crystalline 3C-SiC films on Si (100) and (111) at the reduced temperatures of 800–900°C from the single-source organosilane precursor silacyclobutane ($c\text{-C}_3\text{H}_6\text{SiH}_2$, SCB). In this paper, we report on the chemical vapor deposition of 3C- on 6H-SiC substrates using SCB. SiC growth by SCB has been investigated over the temperature range of 800–1100°C. The growth was carried out at low pressure (5 torr) with 1 sccm of SCB and 1.9 lpm of H_2 . UV and visible spectrophotometry was used to determine an optical absorption edge for the films of ~ 2.27 eV, corresponding approximately to the energy band-gap of 3C-SiC. The structure, strain and dislocation density in the 3C-SiC thin films grown on 6H-SiC are determined using X-ray double crystal diffractometry and compared with values obtained for 3C-SiC grown on Si(111). The SiC films grown by SCB on Si at 900–1000°C were found to be 3C-type crystalline and with an excellent surface morphology. The films grown at 1100°C were found to be a mixture of cubic and hexagonal polytypes of SiC. Although the dislocation density of SiC on Si(111) was in the low to mid- $10^{10}/\text{cm}^2$, which is a considerable improvement over the values present in 3C-SiC films grown on Si(100), an even more significant reduction in the dislocation density has been achieved for 3C-SiC grown on 6H-SiC (0001). These films exhibit a dislocation density in the low to mid- $10^7/\text{cm}^2$, which is within range of the substrate dislocation density.

The results obtained indicate that use of SCB as precursor for growth of 3C- on 6H-SiC is very promising for low temperature deposition of crystalline films.

EM-TuP24 Electron Emission from Wide-Bandgap Negative Electron Affinity Materials, C. Bandis, D. Haggerty, and B. B. Pate, Department of Physics, Washington State University, Pullman, WA 99164.

The diamond (111) 1 \times 1 surface was found to exhibit negative electron affinity (NEA) over a decade ago [1]. Our recent work [2] on electron emission properties of the NEA diamond (111) surfaces find that the creation of free excitons play an important role in the high quantum yield which has been obtained with near threshold photo-excitation. We found that bound electron-hole pairs transport to the surface and breakup in the electric field of the surface dipole. In this report, we present evidence for the existence of such an emission mechanism in other wide bandgap materials. These results suggest that the exciton-derived emission mechanism is common, and is likely to apply to a wide variety of wide-bandgap materials.

[1] F. J. Himpsel et al. Phys. Rev. B20, 624 (1979).

[2] C. Bandis and B. B. Pate, in preparation.

Work supported by the National Science Foundation.

EM-TuP25 Conformality of SiO_2 Films from TEOS-Sourced Remote Microwave Plasma-Enhanced Chemical Vapor Deposition, D. A. Levedakis, H. Liao, T. S. Cale and G. B. Raupp, Department of Chemical, Bio & Materials Engineering, Arizona State University, Tempe, AZ 85287.

The dependence of silicon dioxide step coverage in micron scale trenches on substrate temperature, absorbed microwave power, total pressure and O_2 :TEOS flow ratio were systematically investigated in a remote plasma configuration in which oxygen is excited upstream of the deposition chamber in a microwave discharge. This configuration allows us to investigate the plasma-induced deposition of SiO_2 from TEOS in the absence of charged species (ions and electrons). Of the independent parameters investigated, temperature has the strongest effect on film conformality. For example, step coverage in a long rectangular trench of aspect ratio two is greater than 90% at 250°C., but less than 40% at 400°C at otherwise fixed conditions. Experimental film profiles in trenches are compared to simulations using EVOLVE, a ballistic transport and reaction simulator. In our simulations we assume that deposition proceeds through oxidative attack of TEOS or TEOS fragments adsorbed on the growing film surface by oxygen atoms. A thermally activated, heterogeneous oxygen atom recombination reaction consumes oxygen atoms without leading to deposition. A plasma chemistry model of the excitation and afterglow regions of the plasma source is used to provide an estimate of the flux of oxygen atoms to the feature mouth. We demonstrate how physically-based modeling can be used to extract kinetic rate parameters from experimental data, and how to use physically-based simulation to predict operating windows for high step coverage and good film quality.

MANUFACTURING SCIENCE AND TECHNOLOGY Room BR4 – Session MS-TuP

Manufacturing Science and Technology

Moderator: G. W. Rubloff, North Carolina State University.

MS-TuP1 Low-Cost Optical Reflectivity Temperature Measurement System, H. A. Atwater^(a) and D. S. Gardner^(b), (a) California Institute of Technology, Pasadena, CA 91125 and (b) Intel Corporation, Santa Clara, CA 95052.

We describe the results of a project to assess the use of a low-cost ($\leq \$700.00$) optical reflectivity system as a practical surface temperature diagnostic for on-wafer measurements during sputter deposition. The physical processes which lead to temperature-dependent optical reflectivities are material-dependent, but are large enough for many integrated circuit materials (e.g., Al, Si, Cu) to be useful for thermometry. Correlation of thermocouple temperature measurements made in an isothermal environment with optical reflectivity allows

system calibration via an empirical fit to the nonlinear dependence of reflectivity with temperature. Dividing the reflectivity data for a given material by the fitting function allows relative temperature measurement with precision of $\pm 4^\circ\text{C}$ at temperatures above 170°C , for Al and Si. *In situ* optical reflectivity measurements during sputter deposition of Cu indicated a large temperature rise during film deposition. Limits to optical reflectivity thermometry, as well as extensions to rapid thermal annealing and chemical vapor deposition will be discussed.

MS-TuP2 Magnetron RIE System Using a Dipole-Ring Magnet for Quarter Micron Etch Process, Y. Tahara, Y. Ishikawa, M. Ogasawara, K. Inazawa, Etch System Dept. SPE Div., Tokyo Electron Limited, Sumiyoshi-cho, Fuchu 183 Japan, K. Horioka, H. Hayashi, *Y. Yoshida, H. Okano, Research and Development Center, *Semiconductor Div., Toshiba Corporation, Saiwai-ku, Kawasaki 210 Japan.

A Dipole-ring Magnet (DRM) has a capability of producing high fields up to 600G with excellent uniformity. Its low stray magnetic field outside of the ring eliminates interference between chambers, and makes it suitable for compact new mainframe systems. We have developed new processes for quarter micron oxide and Si trench etching employing magnetron RIE system using the DRM.

Si etch rate was found to increase with magnetic field strength due to higher plasma density in high fields. The 600G DRM enables $\phi 0.25\mu\text{m}$ deep trench etch with a rate of more than $1.2\ \mu\text{m}/\text{min}$, which is twice that of the current magnetron RIE. For the SiO_2 process, a magnetic field around $120\sim 200\text{G}$ was adopted according to the optimization of Vdc, ion flux and F radical concentration variations to field strength, to obtain the best etching performance. Finally, contact hole etch process with high selectivity over poly-Si(50), $\text{Si}_3\text{N}_4(20)$, and Al(40) was accomplished with the 120G DRM using fluorocarbon gas and CO chemistry.

The process technologies with DRM are concentrated in a next generation "UNITY" mainframe supported by an advanced control system.

MS-TuP3 Magnetron RIE Without Charge-up Damage Using a Dipole-Ring Magnet, J. Sakai, S. Ikeda, M. Sekine, K. Horioka, *Y. Yoshida, H. Okano, Research and Development Center, *Semiconductor Div., Toshiba Corporation, Toshiba-cho, Saiwai-ku, Kawasaki 210 Japan, M. Ogasawara, K. Inazawa, Y. Tahara, Etch System Dept. SPE Div., Tokyo Electron Limited, Sumiyoshi-cho, Fuchu 183.

We have introduced a new plasma etching system using a Dipole-ring Magnet (DRM) as a promising etch tool for quarter micron devices. DRM provides a parallel magnetic field up to 600 Gauss with excellent uniformity, and has achieved excellent performances such as high etch rate in Si deep trench and high selectivity of more than ten in SiO_2 etching over Si_3N_4 as well as low charge-up damage (1).

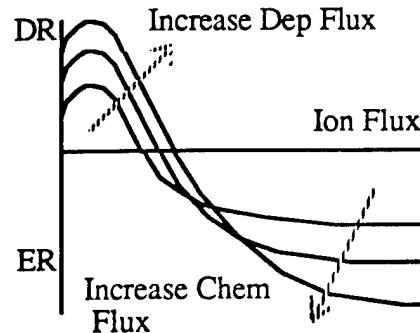
For further improvement of the gate oxide reliability in SiO_2 etching, we have succeeded in compensation of the plasma non-uniformity due to $E \times B$ electron drift, realized by use of an optimized magnetic field distribution. This enabled the use of higher rf power for higher etch rates, and a compact magnet design, still with no degradation of the gate oxide in a TDDB test. Finally, the DRM's low level of stray magnetic field eliminates interference between chambers, which makes it suitable for compact mainframe systems.

(1) M. Sekine et al. Proc. 13th Symp. Dry Process, (Tokyo, 1993) p. 17. (The Inst. of Electrical Engineer of Japan).

MS-TuP4 Profile Modeling of High Density Plasma Oxide Etching, J. S. Han, J. P. McVittie, J. Zheng, Stanford University, Center for Integrated Systems, Stanford, CA 94305.

Plasma dry etching is increasingly becoming a central part of IC manufacturing. As device dimensions continually decrease, the contact vias and lines begin to push the sub $0.35\mu\text{m}$ regime. In addition, aspect ratios approaching 5:1 for contacts and more than 20:1 for trenches are now commonplace. Such geometry can only be processed using dry etching techniques. To understand and predict etch profiles becomes essential to reduce costs in process optimization. In this paper, several profile issues that result from an industry accepted inductively coupled high density plasma oxide etch system are investigated using oxide overhang test structures and vias. The key features observed from the test structures include ion enhanced polymer deposition, etch rate saturation effect, redeposition, direct polymer deposition (CVD polymer), and etch lag. In addition, vias show trends of reverse lag and microtrenching. A new model is presented and implemented in SPEEDIE, a topography simulator developed at Stanford design to use phenomenological models to predict profile evolution. The model

separates the neutral flux into a deposition flux and a chemical flux, where the deposition species contribute to the growth of polymer and the chemical species contribute to the etching of the substrate, in this case, SiO_2 . Deposition or etching takes place at a rate dependent on the ratio of the deposition flux to the ion (energy) flux or chemical flux to the ion (energy) flux. In addition, microtrenching is modeled by an ion induced chemical desorption component. Comparison with SEM results show that the new model can be used to accurately simulate profile evolution.



MS-TuP5 Insitu Monitoring and Control of MBE and MOCVD Growth Using Multi-Wavelength Ellipsometry, John A. Woollam, S. Pittal, B. Johns, and P. He, J. A. Woollam Co., Inc., 650 'J' St. #39, Lincoln, NE 68508, G. N. Maracas, R. Droopad, C. H. Kuo, and S. Anand, Arizona State University, Tempe, AZ 85287, S. Dakshina Murthy and I. Bhat, Rensselaer Polytechnic Institute, Troy, NJ 12180.

In this paper we describe real-time growth control of MBE grown GaAs/AlGaAs multiple quantum well, and MOCVD grown HgCdTe using a novel and low cost insitu multi-wavelength ellipsometer. This ellipsometer acquires insitu ellipsometric data in real-time at 44 wavelengths in the spectral range of 415-750 nm with maximum acquisition rate of 25 measurements/second (typical 1 measurement/second).

A GaAs/AlGaAs multiple quantum well with varying well thicknesses was MBE grown on a GaAs substrate. In this experiment layer thicknesses of the multiple quantum well were controlled by the ellipsometer. The substrate was rotated at all times during the MBE growth. The insitu ellipsometer was also used in the composition control of MOCVD grown HgCdTe. Alloy compositions of 0.25 and 0.3 were stabilized by regulating the H_2 flow through a DMCd source. And, when an intentional temperature disturbance was introduced during the control experiment, the ellipsometer re-adjusted the H_2 flow to maintain the as-grown composition. Results from SIMS will also be described.

MS-TuP6 Electrical Monitoring of Surface Conditions in a Plasma Reactor, Mark A. Sobolewski and James K. Olthoff, National Institute of Standards and Technology, Gaithersburg, MD 20899.

Measurements of the voltage and current of radio-frequency discharges can be used to detect plasma etching endpoints and monitor the conditioning of plasma reactor surfaces. However, the mechanisms that relate plasma electrical characteristics to surface properties are not well understood. We have therefore undertaken a detailed investigation of these mechanisms. In this work, voltage and current waveforms were measured at both electrodes of a parallel-plate reactor as gas composition and surface conditions were varied. To further characterize the electrical properties of the plasma, the rf voltage in the glow region of the plasma was measured, using a capacitive probe. Absolute concentrations of gas-phase species were measured simultaneously using a calibrated mass spectrometer.

Plasma electrical properties can depend indirectly on surface chemistry, through changes in the gas-phase concentration of species arising from the surfaces. We have also observed, for aluminum surfaces exposed to oxidizing plasmas, electrical changes that depend directly on surface conditions. At fixed gas-phase oxygen concentration, slow changes were observed in plasma impedance and dc self bias, due to absorption of oxygen species by the aluminum. The electrical changes arise because the adsorbed oxygen increases secondary electron emission from the aluminum surface, producing greater electron density in the plasma. The electrical changes can be reversed by exposing the surface to an argon plasma, which removes the adsorbed oxygen by sputtering.

We have developed a model of the electrical behavior of rf discharges

that can be used to interpret the data and to distinguish changes in surface conditions from competing effects. The model suggest that electrical sensors of surface conditions would succeed in a wide variety of plasmas. The model also allows values of other relevant plasma properties (such as the plasma potential and incident ion energies) to be obtained from the electrical data.

MS-TuP7 Field Emission Tips for Micro-Column Lithography, *W. K. Lo¹, M. Skvarla¹, M. S. Isaacson²*, ¹Applied and Engineering Physics, Cornell University, Ithaca, NY 14853, ²National Nanofabrication Facility, Cornell University, Ithaca, NY 14853.

We have tailor made field-emission tips with differing geometrical shapes using controlled electrochemical etching in combination with nanofabrication techniques including focused ion beam milling and reactive ion etching. Current investigations include studying the effects that a microfabricated Schottky electrode, formed integrally with the tip, has on the tip's emission angle, extraction voltage, and energy distribution.

These emission properties are all relevant to micro-column based electron lithography¹ which requires electron sources exhibiting: 1) low extraction voltages (~ 1 kV) to accommodate low-voltage microcolumn operation, 2) narrow energy distributions to reduce the effect of chromatic aberration on spot size, 3) narrow emission angles to reduce the effect of spherical aberration on spot size, 4) high current densities to allow faster scanning for a given electron dose, and 5) stable emission to minimize unintended dose variation.

Field-emission sources, best known for their narrow energy distributions and high current densities, have the potential to satisfy all these requirements simultaneously. Conventional sources of this type are, however, notoriously sensitive to contamination and sputter damage by ionized residual gases and usually operate under ultra-high vacuum conditions ($\sim 5 \times 10^{-11}$ Torr) for stability. They also have relatively high extraction voltages (few kV) and emission angles ($\sim 10^\circ$). These properties are affected by the geometry of the tip and the tip material which can be controlled.

Recent results from our study will be presented here.

1. T. H. P. Chang, D. P. Kern and L. P. Muray, *J. Vac. Sci. Technol. B* 10 (1992) 2743.

MS-TuP8 High Resolution Resists for Low Energy Electron Beam Lithography, *C. W. Lo¹, M. J. Rooks², H. G. Craighead, ¹Applied and Engineering Physics, Cornell University, Ithaca, NY 14853, and ²National Nanofabrication Facility, Cornell University, Ithaca, NY 14853.*

Low energy electron beam lithography has been suggested for use in high resolution patterning because of low proximity effects and high resist-sensitivity at low energy. However, resists and electron-beam columns optimized for use in low energy lithography are not yet well developed. Micro-column lithography is one approach which

addresses the difficulties of obtaining a finely focused, low energy electron beam. Using a miniature, micro-fabricated electron column (a few mm in length), scanning electron beams with diameters of tens of nm at 1 keV have been achieved¹. The response of resists to electrons of different energies must be considered for the development of low energy resists. For instance since electrons at low energy have very shallow penetration depths, the resists has to be very thin.(tens of nm for 1 keV electrons). Some resists are capable of high resolution pattern-transferring, but because of their low electron sensitivities at high energies, they are impractical to employ for conventional lithography. These resists are, however, attractive for use in low energy lithography because cross sections for 1 keV electrons are approximately two orders of magnitude higher than those for 30 keV electrons.

We are currently investigating several promising processes for use in low energy, high resolution pattern transferring. The resists are exposed using a low energy, field emission SEM, which has been modified to include a pattern generator. It has been used successfully to expose patterns with 1 keV electrons on PMMA and self-assemble monolayers. In addition, a Monte-Carlo program has also been developed for simulating the non-classical, low energy electron-matter interactions. We will discuss our most recent experimental results which include comparisons to the predictions of Monte-Carlo simulations.

¹Chang et al., *J. Vac. Sci. Technol. B* 10, 2749 (1992).

MS-TuP9 The Design and Testing of a Multi-Task, Multi-Instrument Sample Transfer System, *S. Thevuthasan and D. R. Baer, Pacific Northwest Laboratory, Richland, WA and J. N. Worthington, T. R. Howard, and J. R. Munn, Thermionics Northwest Instruments, Port Townsend, WA.*

A prototype multi-instrument, multi-task UHV sample transfer system has been developed for integration with a wide range of synthesis and analysis instruments. This capability will be utilized in the Environmental Molecular Sciences Laboratory (EMSL), a new U.S. Department of Energy user facility under construction at Pacific Northwest Laboratory (PNL). The EMSL will have several state-of-the-art systems that can be used by scientists from around the world. A primary focus of EMSL is understanding the molecular level interactions that influence waste processing and contaminant transport in the environment. Because interfaces play a large role in these environmental issues, there is a significant element of interface science in EMSL. The specimen transfer capability allows a sample to be synthesized, processed, and characterized by several surface science techniques without exposing the sample to air. The temperature range of the specimen can be as high as 2000 K during heating and as low as 50 K during cooling. Components of the system are designed to allow pumped or ambient transfer of specimens from different locations. Several tests have been performed to verify the functional limitations and capabilities of the system. We will discuss the design and the capabilities of the transfer system along with test results.

layer growth conditions. In addition, they provide effective in-situ monitors of heterointerface quality and stability.

1. R. Nicolini et al., Phys. Rev. Lett. 72, 294 (1994).
2. J. Gutowski, N. Presser, and G. Kudlek, Phys. Stat. Sol.(a) 120, 11 (1990).

9:40 am **EM-WeM5 Materials Issues in II-VI Semiconductor Lasers**, *J. M. DePuydt, S. Guha, M. A. Haase, J. Qiu, G. E. Hofler, B. J. Wu, G. Meis-Haugen and H. Cheng*, 3M Company, St. Paul, MN 55144-1000.

Since the first report of II-VI blue-green laser diodes in 1991 rapid progress has been made in advancing both the II-VI materials and devices. Although continuous operation of laser diodes at room temperature has been demonstrated, the lifetimes of the best devices are limited to a few minutes. TEM, cathodoluminescence and electroluminescence studies have shown that laser degradation proceeds by the evolution of dark defects that are formed in the vicinity of pre-existing defects such as V-shaped stacking faults. The stacking faults responsible for the degradation appear to be formed during the nucleation of the II-VI layers on the GaAs substrate. Possible causes of the stacking faults and efforts to eliminate them will be discussed.

INVITED

10:20 am **EM-WeM7 Heteroepitaxy of Nearly Lattice Matched Compound Semiconductors on Silicon**, *K. J. Bachmann, N. Dietz, S. Fiechter, J. T. Kelliher, H. Castleberry, and G. Wood*, Department of Materials Science and Engineering, North Carolina State University, Raleigh, NC 27695.

The control of the properties of heteroepitaxial films of compound semiconductors on silicon is impeded by surface structure/chemistry and strain effects that result in the generation of defects. In this paper, the low temperature growth of nearly lattice-matched heterostructures of compound semiconductors on silicon is reviewed, separating the study of surface structure and chemistry effects from the problem of strain-induced defect formation. Using GaP on silicon as an example, the growth of heteroepitaxial structures under the conditions of interrupted cycle chemical beam epitaxy is discussed. Excellent selectivity of GaP heteroepitaxy on Si as compared to SiO₂ and SiC covered surface areas is observed. Although selectivity is a desirable aspect in the context of device processing, the impediment of epitaxial overgrowth by residual oxygen and carbon on the silicon surface points to the fundamental importance of surface cleaning and the maintenance of a clean silicon surface during the initial stages of nucleation and GaP growth. The kinetics of heteroepitaxial GaP growth is evaluated on the basis of RBS, AFM, HREM, RHEED and mass spectrometric studies that focus on the evolution of chemical and structural changes during the heating of the initially hydrogen terminated silicon surface in the presence of a phosphorus precursor beam and the subsequent nucleation and sealing stage that establishes a contiguous GaP epilayer on the silicon surface. Also, the heteroepitaxial growth of related nearly lattice-matched II-IV-V₂ compounds and alloys is considered, using the ZnSi_xGe_{1-x}P₂ system as an example.

INVITED

MANUFACTURING SCIENCE AND TECHNOLOGY

Room A110 - Session MS-WeM

Advanced Manufacturing Equipment-B

Moderator: C. B. Whitman, CVC Products, Inc.

8:00 am **MS-WeM0 Long-throw Low-pressure Sputtering Technology for ULSI Devices**, *Yoshiyuki Kadokura, Yuzou Kashimoto, Tetsuji Kiyota, Nobuhiro Motegi, I. Nakayama*, ULVAC Japan, Ltd., Shizuoka, Japan.

The process of metallizing contact/via holes plays a very important role in semiconductor device fabrication. As LSI becomes increasingly high integrated, the size of the contact/via holes shrinks, producing higher aspect ratios. As a result, sufficient bottom coverage cannot be obtained by the conventional sputtering techniques. To solve this problem, a collimated sputtering technique has been investigated recently. Though it has been shown that the bottom coverage is highly improved by using a collimator, the technique has still disadvantage for production, such as generation of particles, degassing from the

collimator, and clogging of the collimator itself. We have developed a new technology, called long-throw low-pressure sputtering technology, for sputtering high-aspect-ratio ULSI devices without employing collimators in the system. The basic feature of our design is to make the distance between target and substrate much longer than that of the conventional sputtering method so that sputtered atoms are incident perpendicular to the wafer, while lowering the discharge pressure by one order of magnitude in deposition (10^{-2} Pa range) so that sputtered atoms are not scattered by gas molecules between the target and the substrate. Our results indicate that this technique has very good potential for applications in high-aspect-ratio ULSI sputtering processes. In this paper, we will describe the basic principle of our design, the unique properties of metal thin films, such as TiN/Ti and Al-alloy formed by our method, and the various advantage of the method as an advanced manufacturing technology for next generation ULSI devices.

8:20 am **MS-WeM1 Polysilicon Gate Etch Linewidth and Profile Control in a 0.25- μ m L_{eff} Logic Technology: Effects and Length Scale of Pattern Loading**, *J. W. Adkisson, S. J. Holmes, C. C. Smolinski, R. P. Mallette, M. C. Cantell, T. A. Knotts*, IBM Microelectronics, Essex Junction, VT 05452.

Faster circuit performance requires tight control of gate linewidth and profile to avoid short-channel reliability failures or speed degradation; to provide this control, the gate etch process must be insensitive to variations in loading. Problematic loading densities include very local (<1 μ m), intermediate (1-20mm) and global (100mm) interactions. Our efforts to quantify and reduce these effects are discussed in this paper.

For this study, we used an integrated three-step gate-etch process in a single wafer RIE tool which included a F-based breakthrough etch, followed by a two-step HBr/Cl₂/O₂ polysilicon etch. Cross-sectional and plan-view SEM and electrical linewidth measurements were used to characterize the etch results.

The optimized process showed similar profiles and negligible offset between nested and isolated lines. For very local effects, the offset between isolated and dense features was increased by the passivation produced in the breakthrough step, the degree of sputtering in the first polysilicon etch step, and the oxygen flow ratio in the final gate-oxide selective step. The local loading effects were further perturbed by the intermediate- and global-scale loadings. The length scale of the intermediate loading effects for the gate etch process was determined by using a variable loading structure positioned next to an array of measurement structures. For the most severe loading changes, significant variations in the isolated features extended over 20mm, while nested lines were almost unaffected. Higher global resist loading was observed to decrease etch bias and increase the isolated-dense offset.

8:40 am **MS-WeM2 Interlayer Dielectrics with Low Dielectric Constant for Multilevel Interconnection**, *R. Aoki, N. Hayasaka*, Y. Nishiyama, H. Miyajima*, Y. Nakasaki* and H. Okano**, Integrated Circuit Advanced Process Eng. Department, Toshiba Corp., *ULSI Research Center, Toshiba Corp.

The high quality insulator with low dielectric constant for interlayer dielectrics is the key technology for future high speed ULSI devices. We realized F doped SiO₂ with low dielectric constant (less than 3.5) and high water absorption resistance. F was incorporated into SiO₂ by adding fluorine contained gases into TEOS/O₃ dual frequency excited plasma.

We studied the CVD mechanism by investigating the plasma chemistry and physics, and we have clarified the role of F on film formation properties (film quality and step coverage). And also the mechanism of reducing dielectric constant due to F addition into SiO₂, which was clarified by using molecular orbital calculation, will be presented.

9:00 am **MS-WeM3 Manufacturing Issues of Electrostatic Chucks**, *D. R. Wright, L. Chen, P. Federlin, K. Forbes*, SEMATECH, Austin, TX 78741.

In the past few years, Electrostatic Chucks (ESCs) have become much more widespread in semiconductor manufacturing equipment. In addition to the elimination of moving parts, ESCs hold the promise to decrease the wafer edge exclusion, that is, to allow more good chips to be made on each wafer. A number of technical, material, and business challenges remain in making ESCs workable and reliable in all tools across the semiconductor factor, or fab.

We will discuss issues of clamping force, clamping and declamping time, and wafer temperature control, describing how they affect design and choice of materials. The effect of these choices on adapting ESCs

to various tools over various temperature ranges will also be discussed. The role of models and test results in accelerating development will be addressed.

Finally, we will list some of the business challenges to implementing ESCs. Despite technical successes, many high-volume fab lines are always reluctant to risk installing new technology, despite promises of improvement. SEMATECH has addressed these issues with Working Groups, which help address standard specifications, study early testing results, and share manufacturing performance data.

INVITED

9:40 am **MS-WeM5 Equipment Engineering Methods for Improvements in Particle and Uniformity Performance during Plasma Processing**, *G. S. Selwyn, M. Dalvie, C. R. Guarnieri and M. Surendra*, IBM Research Division, Yorktown Heights, NY 10598.

Particle contamination and process uniformity are key yield detractors in plasma processing. Often, these issues are viewed as separate problems with separate corrective actions. However, recent results demonstrate that uniformity and contamination problems can be *interlinked* by the formation of locally-disturbed plasma regions called "traps". Traps are induced by changes in the electrode including the electrode topography and the emission of secondary electrons. Traps are characterized by a localized increase in the plasma potential. Wafers, clamp rings, focus rings and wafer patterning induce trap formation. In addition to these causes, we demonstrate for the first time that *changes in materials under the electrode surface also induce traps*, by altering the coupling of rf power into the sheath. Traps influence contamination problems by helping to confine particles. Similarly, traps can influence process uniformity by altering the flux and trajectory of ions to the wafer. The latter effect has strong influence on the wafer edge exclusion, the highly nonuniform region close to edge of the wafer. Typically, product yield is lowest in this region.

We correct this problem through the use of specially-designed buried layers of metal and dielectric to compensate edge effects and topography changes. The results are verified by optical emission studies, uniformity mapping and improvement in particle contamination performance.

INVITED

10:20 am **MS-WeM7 Plasma Process Uniformity in a High Density System: Experiment and Modeling**, *C. R. Guarnieri, M. Surendra, G. S. Selwyn, and M. Dalvie*, IBM T. J. Watson Research Center, Box 218, Yorktown Heights, NY 10598.

In plasma processing of large area substrates, nonuniformities in etching or deposition result in lower yields or a wide distribution of device characteristics. Process uniformity is affected by variations in ion flux, energy, and direction.

Experiments have been carried out in a high density, rf inductively coupled system with a rf capacitively coupled substrate. Results indicate that by spatially varying the rf coupling to the substrate alone, we are able to vary the etch rate uniformity of SiO_2 on blanket wafers from a profile that is higher in the center by $\sim 20\%$ to one which is lower in the center by $\sim 10\%$. The rf coupling impedance is varied by both controlling the shape of the metal electrode and the use of dielectrics. The effect of spatially varying substrate rf coupling is dependent on substrate resistivity and applied frequency. Situations where substrate rf coupling is unintentionally nonuniform, e.g. wafer bowing due to backside cooling gas pressure, are also examined.

Process rate uniformity has been modeled with two dimensional analytic models of the plasma source and rf sheaths, coupled to an equivalent circuit element discretization of the electrode assembly, substrate, plasma source and sheath. Results from the model are in reasonable agreement with experimental measurements, and serve as a useful design tool for electrode assembly design.

10:40 am **MS-WeM8 The Complex Impedance of a Dusty Processing Plasma**, *Weston C. Roth and Robert N. Carlile*, Dept. of Electrical and Computer Engineering, University of Arizona, Tucson AZ 85721.

It is now understood that all plasmas used for processing silicon wafers contain large numbers of particles (dusty plasma) which have the potential to contaminate the wafer being processed. It is the purpose of this paper to examine the complex impedance of a dusty plasma. Specifically, we measured the complex impedance (magnitude and angle) at the fundamental frequency of 13.56 MHz and also at the first, second, and third harmonics as a function of time at the RF input to a modified Tegal MCR-1 etch chamber. The plasmas were derived from argon and also SF_6 . For SF_6 , using a Si wafer on an aluminum electrode, there were rapid variations of all angles at early times, probably indicating initial particle nucleation and growth within

the plasma volume and transport to particle traps at the plasma-sheath interface. Subsequent to this period, the angles changed slowly. For argon, with a silicon wafer on a graphite electrode, there was no initial rapid variation, but only a slow change with time, probably suggesting that particle formation is a surface phenomenon. It may be possible to correlate the slow changes in the impedance angles at long times with particle deposition on the wafer, thus eliminating the need for the common and expensive use of witness wafers.

INVITED

BIOMATERIAL INTERFACES NANO 3/ NANOMETER-SCALE SCIENCE AND TECHNOLOGY

Room A106 - Session BINS-WeM

Artificial Cellular Assemblies

Moderator: D. A. Stenger, Naval Research Laboratory.

8:00 am **BINS-WeM0 Electrical Characterization of Artificial Neuronal Networks**, *J. J. Hickman, K. E. Foster, R. C. Oprison, D. A. Stenger¹, A. E. Shaffner², and J. L. Barker²*, Science Applications International Corporation, McLean, VA 22102; ¹Center for Bio/Molecular Science and Engineering, Naval Research Laboratory, Washington, DC 20375; ²Laboratory for Neurophysiology, BNP, DIR, NINDA, National Institutes of Health, Bethesda, MD 20892.

We are creating *in vitro* circuits of mammalian neurons by controlling their adhesion and neurite outgrowth on artificial surfaces. We are using self-assembled monolayers (SAMs) to control the intrinsic and geometric properties of the culture growth surfaces. The ability to control the surface composition as well as other variables, such as growth media and cell preparation, all play important roles in neuron pattern viability. We have recorded the electro-physiological signals produced by neurons on the artificial surfaces in response to stimuli. The surfaces have been characterized by XPS and contact angle measurements and we are relating the intrinsic properties of the surfaces to the cellular development. We are using what we learn for a more fundamental understanding of neuronal circuit development as well as to develop new algorithms for training neural networks.

8:20 am **BINS-WeM1 Neuronal Cells Cultured on Modified Microelectronic Device Surfaces**, *A. Offenbauer¹, J. Ruhe¹, W. Knoll^{1,2}*, ¹Frontier Research Program, RIKEN, 351-01 Wako-shi, Japan and ²Max-Planck-Institute of Polymer Research, 55128 Mainz, Germany.

The recording of the electrical activity of a large number of neurons in tissue culture over a period of weeks or even months should be very helpful in the understanding of the development and function of biological neuronal networks. Ideally, a method for the detection of the changes in intercellular voltage in such a system should have both high spatial and temporal resolution. One approach could be to record with a fixed electrode array built into the floor of the tissue culture chamber.

We have chosen to detect the electrical signal of the neuron by direct coupling with a field effect transistor. Such a coupling is the first step towards multisite recording in neuronal nets and the development of neuronal network on a microelectronic device surface it is necessary, to control adhesion and outgrowth of neurons on a microscopic scale. To achieve biocompatibility the chemical composition of the surfaces of such devices have to be modified.

Our approach to control the chemical architecture at the interface is to attach ultra thin polymer films to the device surface by using a novel "grafting from" procedure. It could be shown, that the chemical composition of the interface could be tuned in such a way that Purkinje neurons show good adhesion to such a surface and survive and grow on it for weeks.

8:40 am **BINS-WeM2 Using Both Topographic Control and Micropatterned Protein Substrates in Controlling Neuron Extension and Connection in Culture**, *Professor A. S. G. Curtis*, Department of Cell Biology, University of Glasgow G12 8QQ, *Dr. Stephen Britland* and *Professor C. D. W. Wilkinson*, Department of Electronics and Electrical Engineering, University of Glasgow G12 8LT.

We describe techniques of combining topographical guidance and patterning of protein molecules on the surface and other adhesive, non-adhesive and activating and inactivating molecules on surfaces,

cm^{-3}) plateau region extending throughout the film thickness ($1 \mu\text{m}$), most likely due to ^2H -point defect pairing. Deuterium in region (i) begins outdiffusion at 300°C in GaN, but in region (ii) does not commence until $>800^\circ\text{C}$. In implanted samples the 2H redistribution occurs with the same characteristics as the bulk population in plasma-treated material. The thermal stability of the deuterium profiles is much higher than in GaAs. Implications for device structure growth, and comparisons with other semiconductor systems will be discussed.

5:00 pm **EM-WeA10 Dry Etching of GaN and AlGaN, I. Adesida and A. T. Ping**, Center for Compound Semiconductor Microelectronics and Department of Electrical and Computer Engineering, University of Illinois at Urbana-Champaign, Urbana, IL 61801; **M. Asif Khan, D. T. Olson, and J. N. Kuznia**, APA Optics, Inc., Minneapolis, MN 55434.

Gallium Nitride (GaN) and Aluminum Gallium Nitride (AlGaN) are wide, direct bandgap materials with high potential for novel electronic and optical devices. To date, the progress in the growth and characterization of these materials has not been matched by advances in processing both in terms of etching, ohmic contacts, and metallization. In particular, it is known that GaN and AlGaN are not easily etched using wet chemistry. Initial progress in etching of GaN has been recorded using reactive ion etching (RIE) techniques. More work needs to be done in the processing of these materials in order to realize their application potential.

In this paper, we present our results on the dry etching properties of GaN and AlGaN. Both reactive ion etching and chemically assisted ion beam etching (CAIBE) methods have been investigated. Etch rate and etch profile results for RIE in Cl-based and HBr mixtures with H_2 and Ar will be presented. Also, results using CAIBE with Ar beam bombardment in an ambient of Cl_2 will be presented. The effect of temperature on the CAIBE etch rates and profiles will be discussed. High etch rates and anisotropic profiles have been obtained for the various methods. For AlGaN, etch rates do slow down significantly at high temperatures if there is any trace of water vapor in the etching chamber. Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS) are used to study etching-induced changes on etched surfaces.

requires constant human intervention to estimate the over-etch time. The algorithm uses *in situ* process signatures and information from a historical database to compute, in real time, the ideal over-etch time for the current wafer etching within the reactor. Our adaptive process gives a comparable standard deviation (about 10 Angstroms) for the remaining gate oxide without human intervention. Since humans have effectively been removed from the feedback loop the process throughput has improved. The production worthy controller is being installed on two reactors in our MOS line in Orlando, Florida.

3:00 pm **MS-WeA4 Real-Time Process Control Method Using Ellipsometry Applied to Gate Etching, J. T. C. Lee, N. Blayo, A. Grevoz, H. L. Maynard, and D. E. Ibbotson**, AT&T Bell Laboratories, Murray Hill, NJ 07974.

Current trends toward single wafer processing using advanced low pressure, high density plasma sources require rapid etching rates that place ever higher demands on the accuracy of process control algorithms and diagnostic techniques. In particular, etching of multilayer gate stacks for ULSI applications requires an extremely anisotropic and rapid process that exhibits unprecedented selectivity to the gate oxide. The high cost of wafers precludes extensive use of time consuming, destructive post-process diagnostics for process development. Optically based diagnostic techniques such as interferometry, optical emission and ellipsometry offer non-invasive and non-destructive means for obtaining real-time information during etching.

We have discovered that with judicious selection of the probing wavelength, ellipsometry signals remain highly sensitive over patterned features, and thus can be used for real-time control of manufacturing processes. The ability to use ellipsometry over patterned structures was unexpected. In this paper, we present the real-time traces obtained at energies from 1.5 to 4.0 eV during the gate etching step of sub $0.35 \mu\text{m}$ CMOS devices using a helicon plasma source. We quantify the effect of mask patterns and underlying topography on the effective band of "useful" wavelengths, i.e., wavelengths that provide characteristic signatures of the endpoints of the various films being etched.

3:20 pm **MS-WeA5 The Application of Real-Time Optical Monitors to Semiconductor Manufacturing, James A. O'Neill**, IBM Microelectronics, Semiconductor Research and Development Center, Hopewell Junction, NY, 12533.

The manufacture of advanced microelectronic devices is testing the limits of dry processing technology. Emerging technologies such as high density etching plasmas and high pressure chemical vapor deposition systems are being developed to meet the fabrication demands of advanced devices. Also, the utility of conventional processing tools is being extended to reduce development and manufacturing costs. In both efforts, optical sensors have received increased attention as a means to characterize novel technologies and to improve the productivity of existing tools. Optical technologies are particularly appropriate for semiconductor manufacturing applications since they provide a sensitive, non-perturbing means to monitor and control dry processes in real-time. This talk discusses the novel application of two optical sensing techniques to semiconductor device fabrication equipment. First, an infrared-based sensor is employed to control the delivery of condensable feedgases to CVD reactors used to deposit dielectric films. This sensor continuously monitors the flow of reactants to the chamber and reduces the need for test wafers typically used to verify film thickness and dopant concentration. Second, an ultraviolet absorption technique is employed to monitor reactants in a high density plasma reactor used for the selective etching of SiO_2 films. This sensor aids process development by identifying reactor parameters which affect the concentration of species which control etch selectivity.

INVITED

4:00 pm **MS-WeA7 Real-Time Process and Product Diagnostics in RTCVD Using In-Situ Mass Spectroscopic Sampling, L. L. Tedder¹, G. W. Rubloff¹, I. Shareef², M. Anderle², D.-H. Kim², and G. N. Parsons¹; ¹North Carolina State Univ., Raleigh, NC 27695, ²IBM Research, Yorktown Heights, NY 10598.**

Reactive chemical processes play a critical role in semiconductor manufacturing, but concentrations of reactive gas species are rarely monitored during the process, nor used for process control. Mass spectroscopy, already in use as residual gas analysis for contamination control in manufacturing, has been exploited here for rapid real-time sensing of both reactant and product species in single-wafer CVD reactions, including RTCVD polySi from SiH_4 and LPCVD SiO_2 from TEOS. Active mass spectrometric sampling at pressures to 5 Torr is achieved using two-stage differential pumping of a sampling aperture in the exhaust gas stream, permitting response times as short as ~ 5

MANUFACTURING SCIENCE AND TECHNOLOGY Room A110 - Session MS-WeA

Diagnostics, Sensors, and Control

Moderator: L. M. Cecchi, Sandia National Laboratories.

2:00 pm **MS-WeA1 Process Control in Semiconductor Manufacturing, S. W. Butler**, Semiconductor Process and Device Center, Texas Instruments, Dallas, TX 75025.

The goal of this presentation is to propose what level of process control in semiconductor manufacturing is required for current device technology and future device technology. The current level of process control implemented in the industry will be highlighted and areas requiring further development will be suggested. Some of the benefits of process control that Texas Instruments has already experienced will be related. The major concepts of process control will be discussed. The technical need for process control, such as machine aging, will be presented, along with the appropriate control implementation schemes. The basic requirements for control to be possible will also be introduced. The role of metrology, specifically *in situ* vs. *ex situ*, as pertaining to process control methods will be presented. Examples of process control applications in semiconductor manufacturing will assist in explaining the concepts introduced during this presentation. The various control methods will be linked to facilities doing research and development of process control.

2:40 pm **MS-WeA3 A Production Demonstration of Wafer-to-Wafer Plasma Gate Etch Control by Adaptive Real-Time Computation of the Over-Etch Time From In-Situ Process Signals, E. A. Rietman, *S. H. Patel, AT&T Bell Labs., Murray Hill, NJ 07974 and *AT&T Microelectronics, Orlando, FLA 32819.**

We describe an adaptive nonlinear controller for wafer-to-wafer plasma gate etch for 0.9 micron CMOS technology. The typical process

sec to concentration and pressure changes in the reactor. In addition to reactant species, gaseous reaction products have been identified and distinguished from cracking fragments of the reactant through relative intensities of mass fragments as a function of wafer temperature (i.e., reaction rate). For rapid thermal polySi CVD from SiH₄, carried out in ~ 30 sec in the range 500–800°C at 5 Torr in 10%SiH₄/Ar, mass spectra reveal not only the time dependence of reactant (monitored by SiH₂, 30 amu), but also—at higher temperatures—reactant depletion and product generation (from H₂, 2 amu). These results demonstrate a basis for using mass spectroscopy in real-time process and product diagnostics for control.

4:20 pm MS-WeA8 Mass Spectrometric *in situ* Process Monitoring Applied to Silicon Dioxide Electron Cyclotron Resonance Chemical Vapor Deposition, L. M. Williams, Lam Research Corp., Fremont, CA 94538, L. C. Frees, T. Vo, Leybold Inficon Inc., East Syracuse, NY 13057.

Conventional residual gas analyzer systems are not adequate for *in situ* monitoring of processes such as chemical vapor deposition (CVD) which involve reactive species. A closed ion source mass spectrometer (MS) system was installed on an electron cyclotron resonance (ECR) CVD tool for SiO₂ films. A mixture of SiH₄, O₂ and Ar at a total pressure of 0.4 Pa was used for the film deposition. The tool was plasma etch cleaned with NF₃ at 400 Pa. The data collected during deposition suggested that the primary pathway involved the reaction SiH₄ with O₂ to form SiO₂ and H₂, although a lesser amount of H₂O was also observed. Flourine containing residues were detected in the tool after the etch clean. Baking of the MS after sampling an etch clean eliminated the major contribution of MS system flourine residuals to HF and SiF₄ levels detected during SiO₂ deposition. The ability to rapidly and conveniently change the MS ionizer electron energy from the normal 70 eV to 35 eV prevented Ar²⁺ from interfering with HF⁺. Monitoring F⁺ is not a satisfactory way to track HF levels because of the many potential sources of F⁺.

4:40 pm MS-WeA9 Microsensors for Process Control and Monitoring, Robert C. Hughes and James J. Wiczer, Microsensor Dept., Sandia National Labs, Albuquerque, NM 87185.

The Microsensor Dept. at Sandia National Labs is performing Research, Development and Application of microsensor technologies using microelectronic and opto-electronic platforms. The goal is to provide real-time, *in-situ*, inexpensive, multipoint environmental and process control monitoring. Several examples will be described including a new microelectronic hydrogen sensor that accurately measures the partial pressure over 12 decades, from 10⁻⁹ Torr to 100% H₂, surface acoustic wave devices as gas sensors and a fiber optic micromirror for measuring a variety of gas phase species. A multifiber version is being built for NASA/Russian Mars mission to test soil reactivity. Multipoint hydrogen sensors are looking for liquid H₂ leaks at both NASA Stennis and NASA Kennedy. A fully portable system based on the acoustic wave vapor sensor has been fielded as a downhole well monitor. Acoustic wave devices are also finding use in contamination free microelectronics manufacturing. Both regulatory requirements and a desire for cost saving in processing is driving the development and application of microsensor technologies.

INVITED

This work was supported by the US Dept. of Energy under contract DE-AC04-94AL85000.

VACUUM METALLURGY Room A106 – Session VM-WeA

Thin Film Microstructure Evolution

Moderator: L. Hultman, Linköping University, Sweden.

2:00 pm VM-WeA1 Effects of High Flux Low Energy (~ 20 eV) Ion Irradiation During Growth on the Microstructure and Preferred Orientation in TiN Films Deposited by Magnetron Sputtering, I. Petrov and J. E. Greene, Department of Materials Science, University of Illinois, Urbana, IL 61801, and L. Hultman, and J.-E. Sundgren, Department of Physics, Linköping University, S-581 83 Linköping, Sweden.

The effects of incident ion/metal flux ratio J_i/J_{Ti} on the microstructure and texture of TiN films produced by ultra-high vacuum reactive magnetron sputtering have been investigated using x-ray diffraction and cross-sectional transmission electron microscopy (XTEM) including high-resolution XTEM. The films, typically 1.0 μm thick, were deposited at a pressure of 20 mTorr in N₂ on thermally oxidized SiO₂ substrates at 350°C. The flux ratio J_i/J_{Ti} at the substrate was controlled between 1 and 15 by means of a variable axial magnetic field superimposed on the permanent field of the magnetron. The potential difference accelerating the ions to the floating substrates remained relatively constant (~ 20 V) as the ion flux (primarily N₂⁺) was varied. Films deposited at J_i/J_{Ti} = 1 nucleated with a mixed texture, but subsequent film growth resulted in a porous columnar structure in which the preferred orientation evolved to complete (111) texture at film thickness above 0.2 μm . In contrast, films deposited at J_i/J_{Ti} = 14 nucleated with a complete (002) texture which was retained during growth to yield a dense columnar structure. Once film texture fully evolves, however, changing ion flux has little further effect on preferred orientation, as pseudomorphic forces dominate, but still controls intercolumn porosity.

2:20 pm VM-WeA2 The Effect of 20-95 eV Ar Ion Bombardment on Molecular Beam Epitaxy of GaAs(100), J. Mirecki Millunchick and S. A. Barnett, Department of Material Science and Engineering and the Materials Research Center, Northwestern University, Evanston, Illinois, 60208, L. Hultman, Physics Department, Linköping University, Linköping, S-581 83, Sweden.

Low-energy ion-assisted molecular beam epitaxy (IAMBE) is useful for suppressing three-dimensional island nucleation and phase separation in III-V semiconductor alloys. However, little is known about ion damage in this energy range. Defect generation in GaAs(100) has been studied *in situ* by Reflection High Energy Electron Diffraction (RHEED) and *ex situ* by cross-sectional Transmission Electron Microscopy (XTEM). Ar⁺ bombardment was performed at energies E = 20 to 95 eV and current densities J = 0.1 to 0.75 mA/cm² both on static surfaces and during homoepitaxy. RHEED patterns exhibited 2 \times 4 reconstruction and strong Kikuchi lines at all times, indicative of flat and ordered surfaces. XTEM showed that subsurface dislocation loops and stacking faults were generated when E > 50 eV. For example, during bombardment of a static surface at E = 75 eV and J = 0.5 mA/cm², the density of the defects was on the order of 5 \times 10¹⁰ cm⁻². Films grown under 20 < E < 50 eV and 0.25 < J < .75 mA/cm² ion irradiation did not exhibit any defects. While there was no measurable ion etching for E < 50 eV, the deposition rate of films grown at E = 50 eV and J = 0.5 mA/cm², for example, was decreased by 0.16 nm/s. These results show that the beneficial effects of IAMBE can be achieved without causing ion damage or sputtering for 20 < E < 50 eV.

2:40 pm VM-WeA3 Theoretical Considerations on Stress Effects on Thin Film Microstructure, David J. Srolovitz, Dept. of Materials Science & Eng., University of Michigan, Ann Arbor, MI 48109-2136 USA.

Stresses affect the microstructure of thin films in several ways. These include texture effects, island formation, film morphology, and the nature of defects (such as grain boundaries) that make up the microstructure. The microstructure can also play a key role in determining the magnitude of the stresses in a thin film by controlling stress relaxation mechanisms. This talk will review several important areas of stress/microstructure interaction and will focus primarily on how stresses modify texture, grain size, grain boundaries and other planar defects in thin films.

INVITED

3:20 pm VM-WeA5 Surface Thermodynamics Effects on Thin Film Microstructure, R. C. Cammarata, Department of Materials Science and Engineering, The Johns Hopkins University, Baltimore, MD 21218.

Associated with every surface (interface) are surface thermodynamic quantities referred to as surface (interfacial) tension and stress. It is well known how surface tension influences such processes as normal grain growth in thin films. However, there are several other less well known mechanisms of microstructural development where surface thermodynamic properties play a central role.

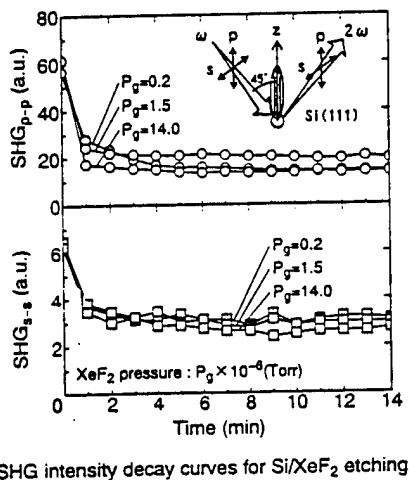
Surface and interfacial tensions and stresses will first be defined and related to thin film systems. This will be followed by a discussion of how these quantities can affect processes involving texture development, epitaxial growth, surface structure, and internal stresses. In particular, the work of Thompson et al. on surface tension effects on

11:00 am EM-ThM9 Observation of Etching Reaction Using Second Harmonic Generation, *S. Haraichi, F. Sasaki, S. Kobayashi, M. Komuro, T. Tani*, Electrotechnical Laboratory, Tsukuba-shi, Ibaraki 305, JAPAN.

The understanding of atomic scale mechanism in dry etching process is very important for the next microfabrication technology. We have investigated the basic characteristics of surface second harmonic generation (SHG) in the Si etching to pursue the possibility of time-resolved observation of etching reaction. A Nd:YAG laser at 1.064 μm was used as the fundamental incident light which was focused on the sample at 45° incident angle. The surface SHG output was detected by a photo-multiplier and a gated integrator. Figure shows SHG_{p-p} and SHG_{s-s} intensity decay curves during the spontaneous etching for Si/XeF₂ system. The suffix of SHG indicates the combination of input-output polarization and the p- and the s-polarization mainly reflect dangling-bond and back-bond dipoles respectively. Both SHG_{p-p} and SHG_{s-s} signals quickly decay with the etching because of the formation of SiF_x layer and approach to the values independent of XeF₂ pressure. Fluorine atom probably does not attack only dangling-bonds but also back-bonds in the time duration observed here and the thickness of SiF_x layer is independent of XeF₂ flux in the steady-state of the etching.

Epitaxial growth of III-V semiconductor materials is probed *in situ* in a molecular beam epitaxy reactor by single photon ionization of the gaseous fluxes using vacuum ultraviolet (VUV) laser radiation. The ninth harmonic of the Nd:YAG laser is used to ionize the species of interest. This radiation is produced by frequency tripling the Nd:YAG laser output to 355 nm and then to 118 nm in a Xe/Ar mixture. Coupled with time-of-flight mass spectrometry, this radiation selectively probes the gaseous fluxes of Ga, As₂, and As₄ during molecular beam epitaxy of III-V materials. The essential aspects of the method and details of calibration procedures to obtain relative fluxes are described. Cracking of the arsenic species does not occur in the laser/mass spectrometer, making relative species concentration measurements very reliable. Rapid data acquisition provides real time measurements of the incident and scattered fluxes and of materials desorbed during growth. Recent work to correlate the flux determinations with Reflection High Energy Electron Diffraction (RHEED) oscillations during GaAs epitaxial growth will be discussed.

[†]Staff Members, Quantum Physics Division, National Institute of Standards and Technology.



11:20 am **EM-ThM10** Hydrogen Annealing Effect on SHG from $\text{SiO}_2/\text{Si}(111)$ Interfaces, *Hiroyuki Hirayama, Fuminori Ito and Kohji Watanabe*, Microelectronics Research Laboratories, NEC Corporation, 34 Miyukigaoka, Tsukuba 305, Japan.

Due to the importance of SiO_2/Si interfaces on MOS devices, many efforts have been reported on the interfacial character. However, SiO_2/Si interfaces are still not fully understood because of the difficulty to probe the interface. With this respect, optical second harmonic generation (SHG) is recently paid attention due to its sensitivity to the interface. But, it is not clear what kind of information on the interfacial character is obtained from SHG. To clarify the origin of the interfacial SHG, especially the effect of interfacial dangling bond, we studied the hydrogen annealing effect on SHG from $\text{SiO}_2/\text{Si}(111)$ interfaces.

In this study, SHG was excited by a 45° incident, s-polarized Nd:YAG laser beam ($\lambda = 1064$ nm). P-polarized SHG from 500A thermally oxidized $\text{SiO}_2/\text{Si}(111)$ interfaces was measured as a function of the azimuthal rotation, after hydrogen- and nitrogen-annealing. Annealing temperature was 430°C in both cases. By fitting the azimuthal rotation dependence of the SHG with a theoretical equation, we obtained nonlinear susceptibility χ_{zxz} and χ_{xxx} for the interfacial SHG. Here, z and x indicate direction along the interface normal and parallel, respectively. We found that χ is decreased only for the hydrogen-annealing. Moreover, the decrease of χ_{zxz} is much larger than that of χ_{xxx} . On the other hand, in our C-V measurement, density of interfacial dangling bond state was found to be decreased only for the hydrogen-annealing. This means that the passivation of the interfacial dangling bond is reflected to the interfacial SHG. The large decrease of χ_{zxz} is caused by the dominance of the interfacial dangling bond along the interface normal direction.

11:40 am EM-ThM11 Single Photon Ionization Time-of-Flight Mass Spectrometric Probing of III-V Semiconductor Growth, *S. M. Casey, [†] A. L. Alstrin, A. K. Kunz, and S. R. Leone, [†] Joint Institute for Laboratory Astrophysics, National Institute of Standards and Technology and University of Colorado, Boulder, CO 80309-0440.*

**MANUFACTURING SCIENCE AND
TECHNOLOGY**
Room A110 - Session MS-ThM

Micro-Contamination and Defects

Moderator: A. C. Diebold, SEMATECH.

MS-ThM1 ABSTRACT NOT AVAILABLE

9:00 am MS-ThM3 An *in situ* XPS Study of Metal Surface Re-contamination and Hollow Cathode Plasma Cleaning, *H. Li, A. Belkand, Z. Orban*, BOC Group Technical Center, Murray Hill, NJ 07974, and *F. Jansen*, Airco Coating Technology, Concord, CA 94524.

Adhesion of coatings depends on the substrate surface condition poor to the coating deposition. Pre-cleaning of metal substrates has been known to improve the coating adhesion; however, little has been published on the quantitative effect of *in situ* plasma cleaning and metal surface recontamination upon exposure to the air.

Using *in situ* XPS, we have studied the recontamination of previously plasma cleaned metal surfaces by exposure to controlled amounts of air. It was found that the initial formation of a thin metal oxide film was followed by the continuing growth of hydrocarbon film as well as hydroxides and moisture from the air. The main source of hydrocarbon is believed to be due to the backstreaming of vacuum pumps. We have evaluated the suitability of a reactive hollow cathode plasma for the removal of the surface contamination. The quantitative reduction of hydrocarbon, moisture, hydroxides and metal oxide films is discussed. A linear plasma source, based on the hollow cathode principle, used for in-line cleaning of metal coil is shown.

9:20 am **MS-ThM4** Generation of Standard Test Wafers for Cleaning Efficiency Evaluations in Semiconductor Manufacturing, *M. Liehr, S. L. Cohen, R. Tsai, K. Pope, B. Furman, R. Purtell, IBM T. J. Watson Research Center, P.O. Box 218, Yorktown Heights, NY 10598, K. Albaugh, S. Basiliere, S. Estes, M. J. Fleming, R. Gaylord, C. Gow, W. Syverson, IBM Microelectronic Division, Essex Junction, VT.*

Wet cleaning processes are ubiquitous in silicon integrated circuit manufacturing. Due to the large consumables cost and potential surface roughening effects there is great interest in evaluation of new and dilute chemical cleans. Thus, it is desirable to use well defined contaminant test wafers so as to enable quantitative comparison of cleaning processes prior to costly evaluations on product wafers. In this study, particle (Si_3N_4 and SiO_2), metal (Fe, Cu, Ni), and hydrocarbon (hexamethyl-di-silazane) standards were prepared and analyzed for use as test wafers for wet cleaning process development. These standard wafers are representative of typical line contamination and at concentrations which are high enough to be a 'worst case' situation. They were designed such that high cleaning efficiency is obtained with a 'good' clean, while for example, water rinsing alone is ineffective for removal. The test wafer preparation and analysis is reproducible cost effective and readily accessible. Using a design of experiments package (BestDesign) which minimizes the number of runs and simultaneously

optimizes multiple responses, it is possible to use this approach for process optimization on manufacturing tools. Requirements for successful implementation of these methods and details of the test wafer preparation/analysis will be described.

9:40 am **MS-ThM5 Rapid Yield Learning, D. M. H. Walker**, Dept. of Computer Science, Texas A&M University, College Station, TX 77843-3112.

Yield is a dominant economic consideration in semiconductor manufacturing. The yield ramp must occur quickly to maximize profits. *Rapid yield learning* is the set of activities directed at increasing the slope of the yield ramp and maintaining high yields once they are achieved.

The basic yield learning cycle is: process a wafer, measure the result, diagnose problems when they occur, and take corrective action. Measurements can be either direct (e.g. optical inspection of a test structure), or indirect (e.g. electrical test of a product chip). Direct measurements can accurately diagnose problems, but are high in cost and low in throughput. They are most useful early in process development, but later become a bottleneck. Indirect measurements are relatively cheap and fast, and are ideal for use during the yield ramp, but require models relating process disturbances to test failures. This has limited their use in the past.

We have developed models relating process disturbances to failures, simulators that implement these models and methods for using them in rapid yield learning. The models must traverse the domains from equipment to process to device to circuit to gate to test. We have used existing and new technology CAD and EDA tools to develop compact, computationally-efficient models that span the domains, providing the defect to failure relationship.

These models have been successfully applied at several companies for diagnosis of parametric and catastrophic defects. Some of these techniques have been commercialized in the *pdFab* simulator from PDF Solutions, Inc. Two challenges have been rapid model characterization and the cultural problems in the use of simulation models in semiconductor manufacturing and test.

INVITED

10:20 am **MS-ThM7 New Ashing Technology with Multi-plasma-mode Reactor, R. L. Bersin, M. Kikuchi, I. Nakayama, ULVAC Technologies, Inc.**, Andover, MA 01810.

Photoresist has been employed in all aspects of wafer processing, such as etching, deposition, and ion implantation etc., to protect a surface from damage caused by highly reactive, or heavily damaging chemical environments to which the wafer is exposed. During the processing, the photoresist mask is subjected to physical and chemical erosion, high temperatures, impregnation with assorted chemical species, surface coating with all sorts of contaminant films and polymers. Considering all of the possible individual processes, and the variety of different chemistries they involve, any manufacturing tool purported to be a stripper of photoresist faces a severe test of flexibility. Historically, photoresist dry stripping equipment has concentrated on the simple chemistry of oxygen plasma, which quite effectively handles the virgin organic resist material. However, this approach ignores completely the complexity of the chemistry associated with the residue problems. It has been the practice of the industry to leave that problem up to the device manufacturer, to develop the necessary complex wet-chemical process steps. We have developed a new process methodology, that combines microwave downstream free-radical chemistry and low-bias ion chemistry in one reactor, and in appropriate processing-step sequences to remove bulk photoresist, leaving behind most residuals converted to a water-soluble state. This suggests that DI Rinser-Dryers could replace most of acid and solvent wet stations. Processes have been developed for all significant masking steps. Questions of substrate loss and damage, undercutting of multiple layers, gate-oxide damage, are all being carefully considered. In this paper we discuss about the reactor concept, and present examples of process results and its advantages.

10:40 am **MS-ThM8 Vapor Phase SiO₂ Etching and Pre-gate Oxide Cleaning in an Integrated Cluster System, Y. Ma, M. L. Green, D. March*, K. Hanson, J. Sapjeta and D. Brasen, AT&T Bell Laboratories, Murray Hill, NJ 07974, *Submicron System, Inc., Allentown, PA 18106.**

Silicon dioxide etching and pre-gate oxide Si surface cleaning with vapor phase processing has been studied in a high vacuum, integrated cluster system. The system contains cleaning/etching and rapid thermal oxidation chambers. The integration enables us to process (pre-gate cleaning and oxide growth) an entire cassette without interruption. The SiO₂ etch rate fluctuates from wafer to wafer. The standard

deviation is less than 10% for a 25 wafer lot etched at a rate of 70 Å/min. The on-wafer uniformity is controlled by the IR lamp configuration with higher non-uniformity at higher process temperature. The non-uniformity of etching across a 5 inch wafer is less than 1% with lamps turned off (i.e. room temperature). Both the non-uniformity and the etch rate fluctuation from wafer to wafer can be compensated by an over-etch process since AFM measurement reveals no added roughness to the Si surface. Particles (>0.2 μm) generation during the process is in the range of 20 per wafer after about 100 Å oxide has been etched off. However, the particles counts can be reduced if the etching is carried out at a higher temperature. We are currently engaged in electrical device characterization studies.

11:00 am **MS-ThM9 Detection and Analysis of Ultra-Small Particles on 8" Unpatterned Si Wafers, C. R. Brundle**, C. R. Brundle and Associates, San Jose, CA 95125, and Y. Uritsky, Applied Materials, Santa Clara, CA 95054.

As the line-widths of IC's get smaller, the allowed size of "adder" particles deposited on Si wafers by the manufacturing tool/process also decreases. Currently it is 0.16 μm as detected by laser scanners which allow process engineers to determine the number of "adder" particles on an unpatterned test wafer and establish whether the process/tool is out of spec.

The laser scanner-generated particle map then becomes the reference co-ordinate system for our subsequent attempts to identify the nature of the particles and, therefore, their origin. Particle identification is usually performed using SEM/EDX (morphology information and elemental composition). Problems with this approach increase as particle size decreases, especially since some "particles" turn out to be thin film and organic/polymeric like. This paper reviews these problems and describes approaches for improved characterization capability. The problems are: the difficulty of relocating very small particles in an SEM on 8" unpatterned wafers (a co-ordinate transfer accuracy and an SEM contrast problem); the inability of EDX to effectively separate particle signal from substrate signal; and the fact that morphology and elemental composition are not always sufficient for a positive ID. The improved approaches described are: improved 8" wafer reference origin description and co-ordinate transform algorithms; improved EDX capability; and evaluation of Auger, SIMS, and FIB as "add-ons" to an SEM/EDX system.

11:20 am **MS-ThM10 The Nature of Copper Precipitation From Dilute HF Solutions on to Si Surfaces, T. S. Sriram, R. Sampson, J. Shyu, W. C. Harris, D. Liu and S. Bill, Digital Equipment Corporation, 77 Reed Road, Hudson, MA 01749.**

It is well known that the presence of Cu and other transition metals on the Si surface prior to oxidation can lead to increased junction leakage and possible loss of gate-oxide integrity¹. This contribution examines the effect of Cu contamination of the HF used in HF-last cleaning processes prior to gate oxidation in deep sub-micron Si CMOS processes. We examined P-type (20–40 Ω-cm) Si [100] wafers exposed to a dilute HF solution which was quantitatively contaminated with Cu. Two concentrations of Cu, 1 ppm and 0.1 ppm were used in this study. The surface of the wafers was examined using TXRF and AFM. It was found that the Cu distribution on the wafer surfaces was non-uniform on both large and small scales. AFM imaging showed that the copper deposited in the form of small circular precipitates on the surface. Some areas of the sample exposed to the higher Cu concentration solution showed the aggregation of smaller precipitates in to large platelets. Subsequent Cu deposition on these platelets appears to occur in a layer-by-layer fashion. The surfaces of these platelets were very smooth (~0.1 nm RMS). No aggregation of platelets was seen in the sample exposed to 0.1 ppm Cu in HF. Diffraction analysis of planar samples from these wafers in the TEM revealed that the precipitates were composed of metallic copper.

1. E. Hsu, H. G. Parks, R. Craigin, S. Tomooka, J. S. Ramberg and R. K. Lowry, "Deposition Characteristics of Transition Metal Contaminants from HF-based Solutions on to Wafer Surfaces," Proc. 2nd Intl. Symp. on Cleaning Technology in Semiconductor Device Manufacturing, Phoenix, AZ, 14–18 Oct. 1992, pp. 170–178.



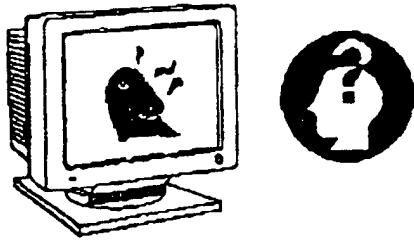
**MANUFACTURING SCIENCE AND
TECHNOLOGY/VACUUM TECHNOLOGY**
Room A110 - Session MSVT-ThA

Vacuum Process Control for Manufacturing
Moderator: W. Weed, Sandia National
Laboratories.

2:00 pm **MSVT-ThA1 Top Ten List of User-Hostile Interface Design**, *Dwight P. Miller*, Sandia National Laboratories, MS 1045, Albuquerque, New Mexico 87185.

This paper describes ten of the most frequent ergonomic problems found in computer-based user-system interfaces (USIs) used in sophisticated industrial machines. In contrast with being "user friendly," many of these machines are considered "user-hostile" by the author. The historical lack of consistent application of ergonomic principles in computer-based USIs has led to a breed of very complex industrial equipment that few people can operate safely and efficiently without extensive orientation, training, and experience. This design oversight has created the need for extensive training programs and help documentation, unnecessary "human" errors and incidents, machine downtime, and reduced productivity resulting from operator stress and confusion. The ten issues are treated in a problem-solution format with real-world graphic examples of good and poor design. Intended for a diverse audience, the paper avoids technical jargon, and is appropriate reading for those involved in software, product engineering, marketing, and management.

INVITED



2:40 pm **MSVT-ThA3 Advanced Control Methodologies Into the Future**, *Richard W. McMahon*, Techware Systems Corporation, 100-Future, 12051 Horseshoe Way, Richmond, BC, V7A 4V4, Canada.

Along with other aspects of vacuum deposition and etch, control system methodologies and practices are undergoing a process of maturation. During the 1980s exciting research was conducted into the use of exotic techniques such as real-time expert systems, artificial neural networks, and fuzzy logic, with the aim of modeling and closing control loops on poorly understood plasma based processes. Currently, researchers are exploring a variety of sensor technologies and a more classical approach to multivariate control, while systems developers direct their efforts towards quality, usability and interoperability issues, reflecting the maturing needs of the deposition and etch industry. A great deal of collaborative work is going into the creation of designs and standards for connecting equipment controls into factory networks and, in the case of cluster tools, to each other. Standards are also evolving for human interfaces, sensor buses, and electronic document retrieval. Software quality improvements such as the separation of applications (customization) code from systems (standard platform) software modules, self diagnostics, version control, and qualification tests are beginning to appear. By selectively drawing upon software technologies available in a wider range of processing industries, vacuum control systems of the future will have improved friendliness, reliability, and integration with other computer systems used throughout the user's facility.

INVITED

3:20 pm **MSVT-ThA5 Adaptive Extensions to a Multi-branch Run-to-Run Controller for Plasma Etching**, *James R. Moyne, Nauman Chaudhry, and Roland Telfeyan*, Univ. of Michigan, DTM Center, Ann Arbor, MI 48109-2108.

Fuzzy logic and database learning mechanisms have been incorporated into a generic plasma etching run-to-run controller, resulting in a very dynamic, adaptable and robust system. The system features an Applied 8300 RIE controlled by a Techware II equipment controller. A TCP/IP connection links this equipment controller to the run-to-run controller residing on a SUN. The run-to-run control environment

is generic in that the basic control framework and controller development results are applicable to VLSI manufacturing in general. The controller is multibranch as it utilizes multiple algorithms in complementary fashion to achieve process optimization and control. The current implementation utilizes three branches: (1) a linear approximation control algorithm, (2) an optimization algorithm that utilizes (real-time) data collected in-situ to determine optimal run-to-run process parameter settings, and (3) a statistical optimization algorithm that utilizes run-to-run data. The controller has been extended to incorporate an automated branch selection process that utilizes fuzzy logic to incorporate process engineer as well as optimization and control algorithmic knowledge. The controller has also been extended to adapt to unforeseen events through utilization of a learning mechanism; this mechanism detects these unforeseen events, intelligently queries the process engineer, guides the engineer through the development of an event servicing scheme, and incorporates this new knowledge into its control knowledge base so that the event may be serviced automatically in the future. Implementation results of the controller (in the control of the etcher) confirm the robust control capabilities in the face of process shift and drift, and recipe change.

3:40 pm **MSVT-ThA6 Real-Time Feedback for Sidewall Profile Control**, *Brian Rashap, Jim Freudenberg and Michael Elta*, University of Michigan.

Real-time feedback control is being applied to the Reactive Ion Etching process as part of a strategy to control sidewall profile. As with a number of etch characteristics, it is not possible to measure sidewall profile in-situ and in real-time. Therefore, an indirect method of controlling these characteristics is necessary. In previous work, we have shown that it is possible to stabilize etch rate by using feedback control to regulate various plasma characteristics.

Currently, we are investigating sidewall profile control, again using feedback to manipulate plasma properties. The sidewall shape produced by an etch is determined by the isotropic and anisotropic etch rate components. Free radicals diffusing to the surface determine isotropic etch rate. Anisotropic etching is caused by an enhanced etch rate in areas exposed to ion bombardment and a reduced rate in areas where a passivation layer is present. This paper reports on the first step in the development of a strategy for controlling sidewall profile. Using a real-time controller for the plasma generation process in a CF₄/O₂ chemistry, a response surface from plasma characteristics (self-bias voltage, pressure, and fluorine concentration) to vertical and horizontal etch rates is developed. A string model simulation, optimized to match scanning electron microscope images of the etch profile, is used to calculate etch rate components. This response surface will then be used to develop trajectories for the plasma environment that produce desired sidewall profiles. Finally, the real-time controller will be employed to actuate these trajectories.

4:00 pm **MSVT-ThA7 Process Monitoring with Residual Gas Analyzers**, *Charles R. Tilford*, National Institute of Standards and Technology, Gaithersburg, MD 20899.

Residual Gas Analyzers (RGAs), most commonly mass spectrometers of the quadrupole type, have long been used for qualitative vacuum-system diagnostics. Increasingly, they are also being used for quantitative process monitoring and control, often involving highly reactive contaminant and process gases. RGAs are complicated instruments and their performance is affected by a number of instrument and vacuum-environment variables, so that even "calibrated" instruments can behave significantly different than expected. The magnitude of the performance anomalies varies for different instruments, but can reach orders of magnitude in extreme cases. This talk will review the important factors affecting instrument performance (including ion-source and quadrupole-filter parameters, total pressure, and exposure to active gases), illustrate the magnitude of observed effects for different instruments, recommend instrument test and calibration procedures, and suggest operating parameters and procedures that can minimize anomalies.

4:20 pm **MSVT-ThA8 An Improved Method of Non-Intrusive Deposition Rate Monitoring by Atomic Absorption Spectroscopy for PVD Processes**, *C. Lu and Y. Guan*, Intelligent Sensor Technology, Inc., 1012A Linda Vista Avenue, Mountain View, CA 94043.

Deposition rate is a critical parameter that needs to be precisely controlled in all PVD processes. Atomic absorption spectroscopy (AAS) is a well established technique for determining the atomic density in vapor phase, and it has been tested for non-intrusive monitoring of deposition rate in PVD processes. However, previous AAS based monitors encountered serious problems during typical deposition processes

due to changes in optical signal levels caused by viewport coating and temperature-induced movements in optical alignment, resulting in poor accuracy for long-term operation. We have developed a novel optical scheme named COPACT (Common Optical Path for Automatic Correction of Transmission) which significantly improves the long-term stability of AAS deposition rate monitors. The emission from a broadband light source, with its central wavelength and bandwidth properly defined by a wavelength selection device, is utilized to compensate the overall optical system transmission change in real-time. This method enables us to fully realize the advantages of AAS for deposition rate monitoring, which include absolute material specificity, high sensitivity, extended vapor sampling region, no background pressure limitation, uninterrupted operation and non-intrusive optical probing. Experimental results are presented to show that it is possible to accurately control very low deposition rates, on the order of one monolayer per minute, over an extended time period.

4:40 pm MSVT-ThA9 A Model Based Technique for Estimation of Fluorine in a CF4/Ar Plasma, P. D. Hanish, Jessy W. Grizzle, and M. D. Giles, University of Michigan, Ann Arbor, MI 48109.

A technique for quantitative interpretation of actinometric data to deduce bulk plasma fluorine concentration in a CF4/Ar plasma has been developed and tested on an RIE. This static, in situ measurement is useful for monitoring fluorine in a manufacturing environment and, in particular, for application of real time feedback control to plasma etching. Based upon a model of CF4 chemistry reaction pathways and products, it improves upon current fluorine estimation techniques by accounting for varying levels of argon dilution resulting from CF4 dissociation. A simple experiment was also developed in order to obtain an estimate of the actinometric scaling factor without an independent measurement of fluorine. Performance of this fluorine estimation technique was compared to that of a standard technique by using time resolved etch rate measurements as an independent indicator of fluorine concentration, while a feedback control scheme decoupled the effects of physical etching by stabilizing the induced dc bias. The model based estimator reduced perturbations in the etch rate by more than 50% compared to those seen when using the standard estimator.

5:00 pm MSVT-ThA10 Role of Inert Carrier Gases in Modeling, Design and Operation of a Single Wafer APCVD Reactor for Manufacturing, Prasad N. Gadgil, Department of Physics, Queen's University, Kingston, Ontario, K7L 3N6, CANADA.

Atmospheric Pressure Chemical Vapor Deposition (APCVD) with inert carrier gases can offer several potential advantages in microelectronic device manufacturing. In a simple stagnation point flow configuration, lower viscosity of inert gases such as Argon and Nitrogen results in a lower pressure drop across the gas distributor and a smaller magnitude of undesirable entrance effects as compared to Hydrogen. In addition, lower thermal conductivity of N₂ leads to the development of isotherms with a higher temperature gradient adjacent to the susceptor surface that are highly desirable to suppress homogeneous gas phase reactions. A hydrodynamic model of a single wafer stagnation point flow reactor is developed. An inverted APCVD stagnation point flow reactor with a novel flow distributor, optimized by flow visualization and fluid flow modeling is designed. The results of its operation with inert carrier gases are described. The suitability of mixing of inert gases with reactive gases such as H₂, NH₃, and O₂ is evaluated for process chemistry of various microelectronic materials. Also, additional economic advantages in the back end of a CVD system in exhaust processing are outlined.

This paper will discuss what is involved in replacing electrolytic hard chrome with modern, clean alternatives. We shall illustrate the discussion with the results thus far of a program designed to evaluate various modern coating technologies as alternatives to hard chrome electroplate on military components. The primary uses of hard chrome in DoD are to hard-coat new components and to rebuild worn parts that come in a wide variety of shapes, sizes, and materials. Any chrome alternative must fit into the total life cycle of the part, which usually includes tear-down, inspection, and recoating several times during its service life.

We shall discuss the uses, capabilities, and performance of thermal spray, laser deposition methods, thin PVD coatings, and plasma nitriding for creating low-wear surfaces at the initial fabrication stage, and for building up worn parts in both military and commercial applications.

INVITED

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2:40 pm VM-ThA3 Development of a Manufacturing Process to Sputter AlN Barrier Layers on MO Recording Disks, David A. Glocker, Eastman Kodak Company, Rochester, NY 14650-2022.

This paper describes the development of an in-line reactive sputtering process to deposit AlN films on polycarbonate disks as barrier layers for magneto-optical recording media. Manufacturing requirements were for an equivalent static deposition rate of at least 2 nm/s and a temperature rise of less than 40°C for the free-standing substrates. The 100 nm thick films had to have low absorption and an index of refraction close to 2.00, as well as be able to provide corrosion protection for the active TbFeCo layer. A dc rather than rf process was chosen in order to achieve the rates and low substrate temperatures needed. Factorial experiments were done to study the dependence of power, total pressure, and the Ar/N₂ partial pressure ratio on the deposition process and film properties. Differences in substrate heating were found that were related to the nature of the dc supply used. Methods of arc suppression were also studied. A dark-space shield that confined the plasma to the racetrack region significantly reduced the number of arcs. However, we concluded that any arcing was unacceptable because it would eventually lead to a loss of control. In order to eliminate arcs completely, we used a 40 kHz power supply driving two cathodes out of phase with one-another. Several methods of process control were compared. Two of them—either operating the target at constant power and using the target voltage to control the reactive gas flow or using an RGA to control the partial pressure of the reactive gas—produced adequate results as demonstrated by control charts of the thickness and optical properties. The effects of target wear on process drift were measured as well.

INVITED

3:20 pm VM-ThA5 Simultaneous Deposition and Lamination Process in Vacuum, A. W. Freeland, J. R. Germundson, R. L. Swisher, K. Barnes and C. T. Wan, Sheldahl Inc., Northfield, MN 55057.

Laminating a protective layer onto a freshly deposited film on a web in situ in a vacuum chamber is very useful in many applications where the deposited film must be protected from scratches or reacting with the atmosphere when it is removed from the vacuum chamber. There are materials that will oxidize in the atmosphere very quickly resulting in undesirable changes in the film's properties. When the laminated protective overlay is used, the material can be protected until it is further processed or put in final use. The process combining the deposition and lamination in one pump-down involves the evaporation of highly reactive material such as bismuth onto a polycarbonate substrate on a roll-to-roll system. Immediately following the deposition, the coated substrate moves to a section of the vacuum chamber where a heat sealable overlay film is bonded to the coated substrate and then rewound onto a take-up roll. A description of such a process and equipment will be presented. A comparison of the film properties with and without the in situ lamination will be presented.

3:40 pm VM-ThA6 Anode Effects in Magnetron Sputtering, A. Belkind, F. Jansen, Z. Orban, and J. Vossen, The BOC Group Technical Center, Murray Hill, NJ 07974.

The anode size and their spatial distribution affect the magnetron discharge characteristics and the deposition rate distribution. Reducing the anode size requires an increased voltage to maintain the same cathode current. Although the magnetron plasma is confined in the race track area, changing the position of small anodes redistributes the plasma density and changes the deposition rate distribution. Applying additional voltage between multiple small anodes allows one to vary the deposition rate distribution in a desired way. The potential

VACUUM METALLURGY Room A106 – Session VM-ThA

Manufacturing Technology for Coatings

Moderator: D. C. Carmichael, Vacuum Technology Inc.

2:00 pm VM-ThA1 Environmentally Compatible Coating Technology[†], Keith O. Legg, A. Adamski, C. West, P. Rudnick, F. Rastagari^{*}, J. Schell^{}, A. Gonzales[†], B. Sartwell^{††}, BIRL Industrial Research Lab., Northwestern University, Evanston, IL; ^{*}Cummins Piston Ring Division; ^{**}GE Aircraft Engines; [†]Corpus Christi Army Depot; ^{††}Naval Research Lab.**

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